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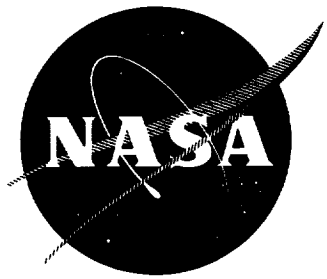

FINAL REPORT
INVESTIGATION OF SMOOTH-BONDED METAL LINERS FOR GLASS FIBER FILAMENT-WOUND PRESSURE VESSELS

by
J.M. Toth, Jr. and D.J. Soltysiak

Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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Douglas Aircraft Company, Inc.
Missile and Space Systems Division
Santa Monica, California

FOREWORD

This report was prepared by the Douglas Aircraft Company, Inc., Missile and Space Systems Division, under NASA Contract No. NAS 3-6293. This project was initiated by Lewis Research Center of NASA to investigate the use of thin-metal liners in glass-fiber, filament-wound, pressure vessels for an operating range from ambient to cryogenic temperatures. The work was administered under the direction of the Chemical Rockets Division, with James R. Faddoul acting as Program Manager.

The report covers work conducted from June 22, 1965 through Feb. 15, 1967. It is submitted in partial fulfillment of the contract and is catalogued by Douglas as Report No. DAC-60640.

J. L. Waisman, Director of Research and Development, and his predecessor, R. W. Hallet, Jr., together with H. H. Dixon and C. Y. Kam provided technical direction. Included among those who participated in the project were J. M. Toth, Jr., Project Director; R. J. Nebesar and D. J. Soltysiak, Research and Development; H. M. Doyle, R. W. Hunter, A. C. Rawuka, G. D. Shepard, W. C. Sherman, and F. M. Tokirio, Materials Research and Production Methods; C. A. Ludwa and R. Yeaman, Engineering Laboratories and Services; and D. W. Yockey, Reliability Assurance.

CONTENTS

FIGURES

TABLES

INVESTIGATION OF SMOOTH-BONDED METAL LINERS FOR GLASS-FIBER, FILAMENT-WOUND, PRESSURE VESSELS

by J. M. Toth, Jr., and D. J. Soltysiak

SUMMARY

Filament-wound fiberglass has been recognized for some time as a potential structure for storing fluids under pressure in a cryogenic environment. The highest potential is realized with a thin, smooth-bonded, metallic liner. The number of cycles that can be achieved with such a liner, however, is dependent upon the ability of the adhesive to prevent the liner from buckling and upon the ability of the liner to resist fracture when subjected to high, plastic, tensile-compressive strains.

The goal of this investigation was the development of a liner-adhesive system, which when incorporated into a vessel, would withstand repeated cyclic loadings over a temperature range of +75°F to -423°F.

Various adhesive systems were evaluated in preliminary coupon testing. A blended polyurethane:epoxy (70:30 pbw) resin with a glass scrim cloth system was selected for further testing in a 1:1 biaxial pressure vessel. A thin aluminum liner performed well (100 pressure cycles to 2% strain) with the adhesive at -423°F, but at ambient temperature, aluminum liners and a nickel liner buckled and failed. On the basis of these results, a reorientation was made to further develop an adhesive system to satisfactorily bond the liner at both ambient temperature and -423°F. The use of a thin nylon scrim in the adhesive, in place of the glass scrim, improved the ambient temperature performance of the 70:30 blend adhesive, while at the same time, it did not cause a degradation in the -423°F performance (10 pressure cycles to 2% strain; this was with aluminum liners; nickel work was discontinued). An 80:20 blend adhesive (with nylon scrim) also performed satisfactorily. The primary liner in all cases remained satisfactorily bonded to the structural wall. However, at -423°F, in all cases, leakage occurred through the bonded longitudinal seam. The test vessels were cycled 5 times to 2% strain at ambient temperature and 10 times to 2% strain at -423°F.

INTRODUCTION

Filament-wound fiberglass has been recognized for some time as a potential structure for cryogenic pressure vessels. However, many problem areas have developed in the evaluation of this potential. The principal one is the containment of the fluid in the vessel over desired pressurization cycles.

Metallic liners seem to be the most promising for full exploitation of the potential fiberglass structure (ref. 1). The major drawback of metal, however, is its low (1/4 to 1/2%) elastic strain compared to the working strain capability of 2 to 2-1/2% in the fiberglass composite. When metal is used as a smooth-bonded liner in a vessel, the liner deforms elastically 1/4 to 1/2% and plastically 1-3/4 to 1-1/2% to match a 2% composite strain upon pressurization. On depressurization, the liner recovers the 1/4 to 1/2% strain elastically, and three possible situations may occur:

(1) The bond remains satisfactory and the liner undergoes plastic deformation without failure so that the structure may be subjected to further pressurizations.

(2) The bond remains satisfactory, but the liner fails as a result of high plastic strains.

(3) The liner buckles and fails at points of high deformation.

The goal of this program was the development of a liner and adhesive system which would withstand repeated cyclic loading, limited only by vessel failure, over a temperature range of +75°F to -423°F. An adhesive system is defined as a combination of adhesive resin and fabric scrim. The work was divided into three tasks.

In Task I, six adhesive systems were evaluated by coupon testing. Data on drum-peel strength, shear strength, uniaxial tensile strength, and thermal contraction of the adhesive systems were obtained at +75°F, -320°F, and -423°F. From these data, a polyurethane/epoxy/glass scrim-cloth adhesive system was chosen for use in the fabrication of the Task II production vessels. A polyester adhesive, with and without scrim cloth was also subsequently evaluated by the Task I tests. An additional evaluation was also made of two polyurethane/epoxy and the polyester adhesive systems.

In Task II, which was performed concurrently with Task I, a subscale-pressure vessel (7-1/2-in. diam by 20-in. long) was designed to achieve 2.0% strain with a longitudinal to circumferential strain ratio of 1/1 in the test section. The fabrication and test of two vessels confirmed the design strain ratio.

There were 22 subscale pressure vessels of the approved design, 11 each with aluminum and nickel liners, scheduled for fabrication. Because of delays in procurement of nickel liners and the promising results obtained with aluminum liners, only two nickel-lined vessels were fabricated. The remainder of the program was redefined and a total of 21 aluminum-lined vessels was fabricated.

In Task III, selected, subscale, pressure vessels of Task II were burst-tested at ambient and cyrogenic temperatures. Other Task II vessels were tested at ambient and cryogenic temperatures by cycling to 2% strain 100 times or until failure occurred. With the realignment of vessel fabrication, the testing was also redirected to evaluate selected adhesives in greater depth. Each of the latter vessels was cycled at both ambient and cryogenic temperatures.

The materials chosen for investigation in this program were all commercially available (with the exception of the Douglas-blended adhesives) and were not necessarily intended for use at cryogenic temperatures. Their acceptance for this use, therefore, is no reflection on their suitability for use as intended by the manufacturer.

PROGRAM

General

Two main problems are encountered in containing liquids or gases at cryogenic temperatures under pressure in glass-fiber, filament-wound, vessels. The first is the result of combining two highly dissimilar materials. Even though the glass-fiber composite is structurally efficient because of its high-modulus, high-strength reinforcement and its low-modulus, high-elongation matrix, neither reinforcement nor matrix could meet the design criteria separately. Glass-fiber composites in a pressure vessel require additional material as a nonstructural liner, bladder, or combination thereof, for containing the fluid or gas because epoxy-resin matrixes craze and crack under composite strains of 1/2 to 2% . Kies (ref. 1) has demonstrated that resin strains are from 3 to 20 times that of composite strains, depending upon resin content at the point of interest. Therefore, the liner must bridge these cracks and discontinuities to provide a barrier to the contained medium.

The other major problem is material embrittlement at cryogenic temperatures. At ambient temperature, the matrix exhibits some flexibility, but when it is chilled to cryogenic temperatures, thermal stresses alone cause cracking and crazing. The adhesive which bonds the liner to the wall also becomes embrittled. The smooth-bonded metallic liners considered for this program (aluminum foil and electroformed nickel) do not exhibit embrittlement at cryogenic temperatures. However, the major drawback of metal is the low (1/4 to 1/2%) elastic strain compared to the working strain capability of the glass-fiber composite (2 to 2-1/2%).

Work on Contract No. NAS 3-2562 (ref. 2) showed that a metallic-lined specimen could be cycled 250 times to 0.8% strain at -423°F before failure occurred. However, during earlier testing, smaller strains at ambient temperature did cause the liner to buckle from the wall because of the greater flexibility of the polyurethane adhesive system at ambient temperature, as compared to -423°F, with a resultant liner failure upon subsequent pressurization.

Therefore, three areas of major concern were:

(1) What adhesive properties are needed to keep the liner bonded to the structural wall at +75°, -320°, and -423°F? What were the potential adhesives and what tests would permit their proper evaluation as candidate materials?

(2) What mechanical properties and hysteresis behavior would the candidate metallic liners exhibit when subjected to the high strains of a glass-fiber composite? What tests would permit the establishment of needed parameters?

(3) What correlation would exist between the test results for (1) and (2) above and the actual behavior in a subscale pressure vessel at +75°, -320°, and -423°F?

The resolution of the above questions comprised the development program.

Task I - Adhesive Evaluation

The adhesive in the composite glass-fiber structure must perform one basic function: it must prevent the metal liner from buckling during depressurization. To achieve this, it must strain with the composite without losing adhesion to either surface or failing itself. The ability of an adhesive to wet the surfaces of the adherends is a principal factor determining adhesive strength.

Properties. - The adhesive properties deemed necessary for successful use were the following:

(1) Strength and wetting ability.

(2) Rigidity.

(3) Toughness.

(4) Coefficient of contraction.

Strength and wetting ability: The present concern was the evaluation of structural adhesives suitable for use from ambient to cryogenic temperatures. As materials are cooled, the internal motion of molecules, atoms, and electrons decreases, which changes physical and mechanical properties. A structural adhesive is usually a complex combination of materials rather than a single substance. In addition, the adhesive is only one component of the composite structure. The overall composite structure, produced from many materials having different properties at cryogenic temperatures, must perform as a unit to be useful. The primary requirement for a structural adhesive is for the adhesive to bond the metal liner to the composite without failing on either surface or within itself. The lap shear test (all tests mentioned in this section will be detailed later) is an excellent method of screening candidate adhesives because it compares adhesive-adherend and cohesive failures with tensile-load information.

Rigidity: Adequate adhesive rigidity is necessary to fully restrain the liner during the decompression cycle. A polyurethane adhesive used in a previous investigation (NAS 3-2562, ref. 2) performed well during testing at -423°F; however, adhesive rigidity was so low at ambient temperature that little restraining force was imparted to keep the liners planar during decompression. As a result, liners buckled and subsequently failed.

Cloth scrim was chosen for the program principally to impart rigidity to the bond line. An important advantage of reinforcing an adhesive with a cloth is that it protects the fluid adhesive during winding. During filament winding on catalized but wet paste adhesives, the pastes are extruded ahead of the incoming glass ends and very little adhesive remains (fig. 1). With a cloth-reinforced adhesive, the fluid pastes are contained in the spaces between the fiber reinforcement (fig. 2). Besides protecting the fluid adhesive, the cloth also acts as a spacer and ensures a uniform glue line.

Two types of fabric were investigated, glass and nylon. The glass cloth is No. 112 with an A-1100 silane finish*. The fabric represents a compromise of weight, openness, and strength. It has low weight with adequate strength. The openness of the weave allows adequate adhesive resin penetration. The initial nylon chosen for consideration was an open-weave cloth, A-2951**. Previous Douglas in-house investigations had demonstrated the applicability of nylon as reinforcement for the adhesive resin.

Toughness: Toughness is the energy-absorbing capability that permits an adhesive to resist further failure after it begins. Because toughness is indicated by the area under the stress-strain curve, a tough adhesive exhibits good extensibility as well as relatively good tensile strength (fig. 3). High tensile strength without good extensibility denotes low toughness (fig. 4). Toughness in this program was determined by uniaxial tensile tests and drum-peel tests.

The uniaxial test indicates potential or "latent" toughness.

The drum-peel test indicates "active" toughness. Peel strength is the resistance of an adhesive system to further failure. "Further" implies that some failure has occurred and that additional failure will follow. An adhesive with high peel strength possesses high residual resistance to further failure even though it may already contain cracks, voids, bubbles, or defects. Moreover, if this same adhesive is subjected to any loading, such as static, dynamic, creep, or fatigue (or alternating expansion or contraction), then these previous failures would not be expected to enlarge progressively until total destruction occurs. A tough adhesive (one with high peel strength) can absorb and distribute large stress concentrations evenly over a wide area.

Coefficient of contraction: A compatible coefficient of contraction with the liner and glass-fiber composite is necessary to minimize differential contraction stresses. Most polymeric adhesives have high rates of contraction which can be considerably reduced by the scrim-cloth reinforcement.

*Supplied by Trevano Glass Fabrics
**Supplied by Stern and Stern Textiles

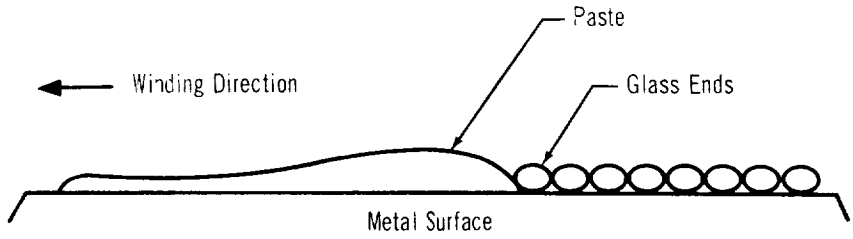


Figure 1. Adhesive without Reinforcement

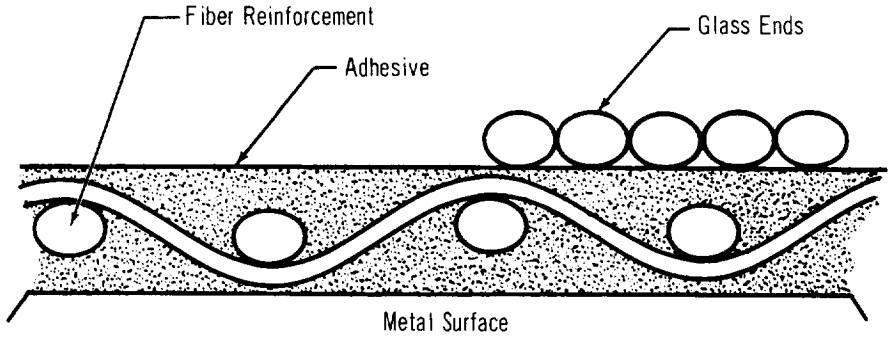


Figure 2. Adhesive with Reinforcement

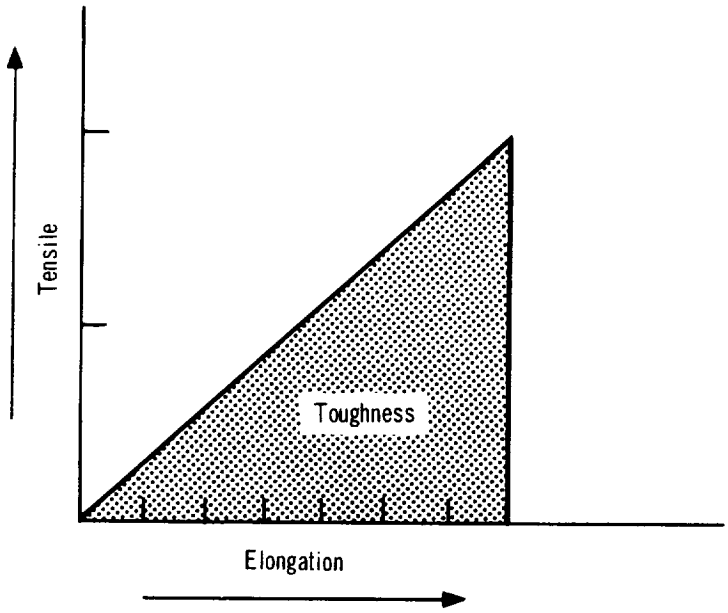


Figure 3. Strain Energy–High Toughness

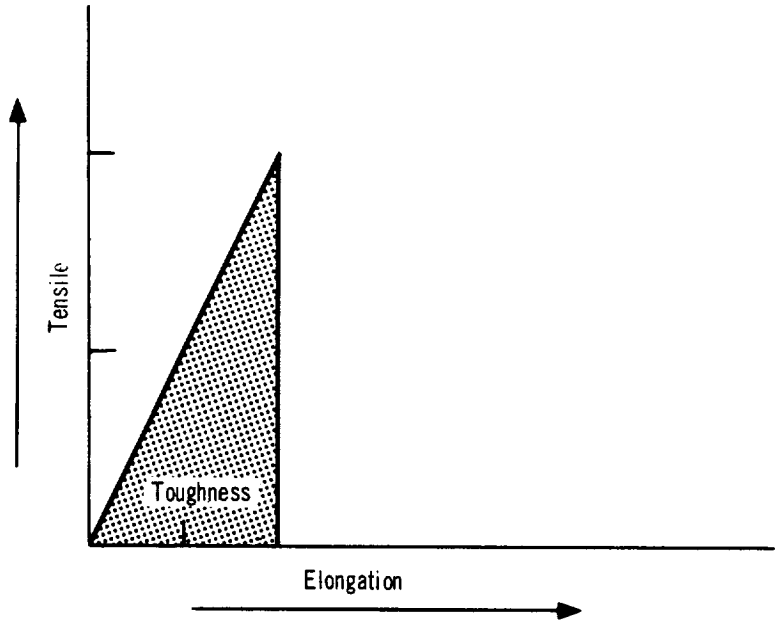


Figure 4. Strain Energy–Low Toughness

Selected Adhesives. - The following four adhesive resins were chosen for evaluation:

(1) Epoxy.

(2) Polyurethane.

(3) Blended polyurethane-epoxy.

(4) Nylon epoxy.

The specific resins are given in table I

TABLE I

RESINS

Adhesive resin	Base	Catalyst	Type
1	Epi-Rez 5101[a]	APCo 322[b]	Epoxy-amine, thermosetting, paste
2	Adiprene L-100[c]	MOCA[c]	Polyurethane prepolymer (polyether type) Diamine, ambient-temperature-curing paste
3	Adiprene L-100/[d] Epi-Rez 5101 (70/30)	MOCA	Mixture of polyurethane and epoxy, heat-curing paste
4	FM 1025[e]	(f)	Heat-curing nylon-epoxy film reinforced with Dacron fabric

[a]Manufactured by Jones - Dabney Co.
[b]Manufactured by Applied Plastics Corp.
[c]Manufactured by E. I. Du Pont de Nemours.
[d]Compounded by Douglas.
[e]Manufactured by American Cyanamid Corp.
[f]Prepackaged adhesive system with catalyst.

The Epi-Rez 5101 system incorporated a nylon scrim cloth; the Adiprene L-100 was tested with both fiber glass and nylon scrim cloths; the polyurethane-epoxy was tested with both fiber glass and nylon scrim cloths; and the FM 1025 system incorporated a nylon scrim. These combinations resulted in a total of six adhesive systems that were evaluated with both aluminum and nickel adherends.

In arriving at the above choices, the following basic adhesives were considered:

(1) Epoxy.
(2) Epoxy phenolic.
(3) Nylon epoxy.
(4) Epoxy polyamide.
(5) Polyamide.
(6) Polyimide.
(7) Polyurethane.
(8) Polyurethane epoxy.
(9) Polyester.
(10) Phenolic.
(11) Nitrile phenolic.
(12) Silicone.
(13) Synthesis of adhesives.

Details of the basic formulations of the chosen adhesives are discussed in the following paragraphs.

Epoxy: Unmodified epoxies have had little cryogenic application because of their inherent brittleness and corresponding loss of flexibility at cryogenic temperatures. Only by modification with various fillers have they had low-temperature applications. However, Douglas research data indicate that the incorporation of a carrier fabric into an epoxy (unmodified by a filler) increases its peel strength and reduces its thermal contraction which thus brings the thermal contraction in line with those of aluminum and nickel (ref. 3). Epoxy resin reinforced with unidirectional glass rovings is characterized by a thermal contraction (+75°F to -423°F) in the thickness direction four times that in the roving direction (ref. 4). These data draw attention to the need for exceptional flexibility and toughness within the adhesive system. The epoxy resin system chosen for the program, Epi-Rez/APCo 322, exhibits extremely high strength in acompposite with E-HTS Fiberglas (ref. 5). Epi-Rez 5101 is a highly refined bisphenyl A epoxy resin and APCo 322 is an aromatic amine hardener. To give added continuity to the complete vessel, this resin system was evaluated for application as the liner adhesive, because it was also being used as the composite matrix.

Polyurethane: In May 1963, the final report of a NASA-sponsored study for the development of low-temperature adhesives was published (ref. 6). This study was one of the first efforts to formulate adhesives specifically for cryogenic use. The most significant result of the program was the development of an adhesive which increased in strength as the temperature decreased. The adhesive was a two-part system composed of urethane resin and an aromatic amine hardener, and it is produced commercially as Narmco 7343/7139*. The material was not new, but its use as an adhesive was. Components of the material were produced by DuPont and called Adiprene L-100, and the catalyst was MOCA (methylene orthochloroaniline).

* Manufactured by Narmco Materials Division, Whittaker Corporation.

Cryogenic fatigue studies performed by Douglas (refs. 2 and 7) substantiate the exceptional fatigue strength of the polyurethanes.

Polyurethane-epoxy: The urethane resin, Adiprene L-100 was selected for blending with an epoxy resin. The epoxy resin was Epi-Rez 5101 to minimize the variables in the composite system.

Studies at Du Pont (ref. 8) indicated that as the proportion of epoxy resin in the urethane polymer increases, the hardness, tensile strength, and flexural strength increase. But this increase produces a decrease in the polymer's impact strength, resistance to thermal shock, and percent elongation. Therefore, this modification increases the strength of the polyurethane elastomer at the expense of flexibility. This added strength will be most apparent at room temperature where polyurethanes are traditionally weak. A 70:30 (parts by weight) mixture was selected for the initial blend ratio.

Nylon epoxy: The relatively new nylon-epoxy adhesives represent a mixture of a thermosetting resin (epoxy) and a thermoplastic resin (nylon). In most nylon-epoxy structural adhesive films, the nylon acts as a matrix for the epoxy resin. When the adhesive is heated under pressure, the nylon melts and wets the surface. The epoxy resin is cured by a system that becomes active at the prescribed temperature. The relatively hard epoxy resin system, in effect, reinforces the softer nylon matrix. An example of this is FM 1025. The nylon-epoxy adhesives are tough and one of the few systems which has both high tensile lap-shear and high peel strength.

Method of Investigation. - The best adhesive systems for each liner were to be determined by screening the six selected adhesive systems in the following four-phase test program:

(1) First test phase--The six adhesive systems were to be evaluated in tensile lap-shear tests at +75°F, -320°F and -423°F, to determine the adhesion characteristics of the adhesive to the liner-composite and the cohesive strength of the adhesive.

(2) Second test phase--Concurrently with the tensile lap-shear tests, the six adhesive systems were to be evaluated at +75°F, -320°F and -423°F in a drum-peel test configuration, which simulated as nearly as possible actual conditions in a filament-wound pressure vessel.

(3) Third test phase--From the first two phases, the three best adhesive systems for each liner were to be evaluated for thermal contraction characteristics. Coefficient-of-contraction tests were to be made on each selected adhesive system from +75°F to -103°F, -320°F, and -423°F.

(4) Fourth test phase--Uniaxial tensile mechanical properties tests were scheduled for the three best adhesive systems for each liner. Testing was to be done at +75°F, -302°F, and -423°F.

Tensile-lap shear testing: The test specimen consisted of three components: the metallic adherends, the adhesive system, and the preimpregnated fiber-glass tape. The components were arranged as shown in figure 5.

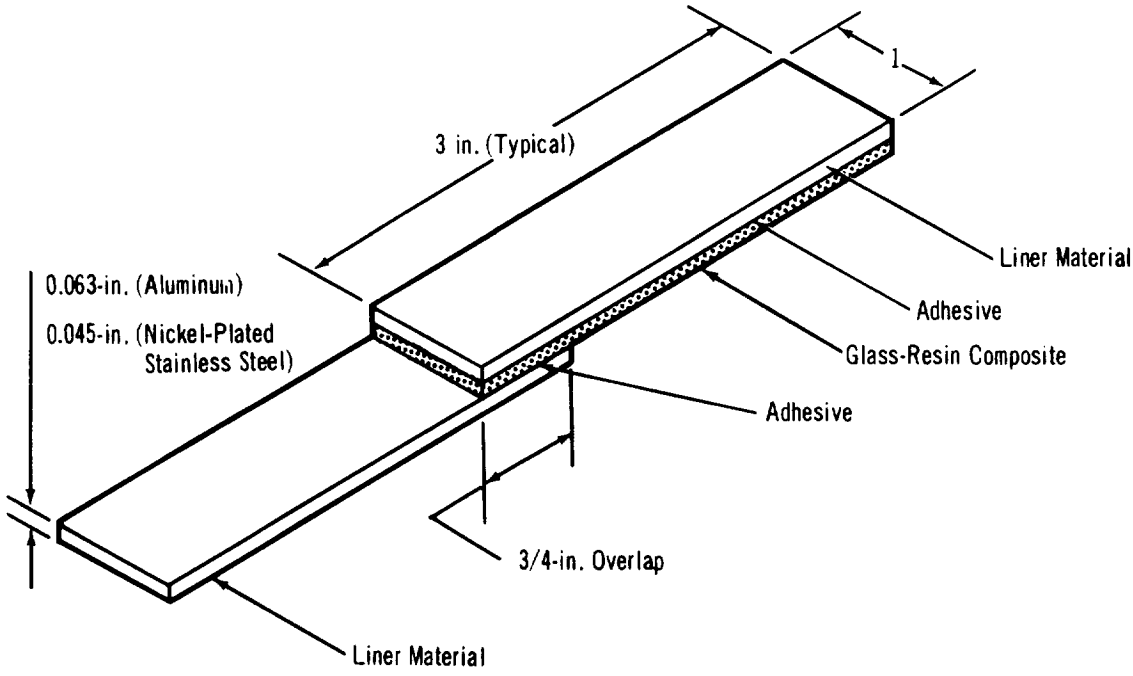


Figure 5. Adhesive Tensile Shear Test Specimen

The specimen was constructed to represent as closely as possible the bonded structure of the filament-wound pressure vessel. The bond overlap was lengthened to 3/4 in. from the standard 1/2 in. called for in ASTM D 1002-64T. The inclusion of a fabric within the bond line, plus the introduction of the epoxy-glass composite surface as one of the adherends, complicated the 1/2 in. overlap area to the extent that an increase in the bonding area was deemed necessary to provide an accurate representation of the bonded structure. The composite surface, which was used as one of the adherends, was prepared from two plies of preimpregnated tape, which had been bonded to a metal coupon.

Detailed specimen processing is described in Appendix A.

Five specimens of each candidate adhesive system with each liner material were tested at each of the three test temperatures (+75°F, -320°F, and -423°F). Results are shown in figures 6 and 7.

The specimens were mounted in a universal loading machine with steel pins. The cryogenic tests were performed in a cryostat mounted on the testing machine. The specimens were soaked until thermal equilibrium had been achieved (minimum specified time of 10 min.). Testing was performed at a load rate of 600 to 700 lb/min. at all three temperatures.

From the data shown in figures 6 and 7, the following conclusions can be drawn. The shear strength of the polyurethane systems increased at cryogenic temperatures for both nickel and aluminum adherends. The shear strength of the epoxy systems remained approximately constant or decreased at cryogenic temperatures for aluminum adherends. The addition of epoxy to the polyurethane system increased the shear strength at room temperature, as intended, for both the nickel and aluminum adherends. The polyurethane/epoxy mixture also exhibited higher shear strength at cryogenic temperatures than the polyurethane system with both nickel and aluminum adherends. Shear strength of the adhesive systems with aluminum adherends was higher than those obtained with nickel, indicating better adherence to the aluminum.

No valid results were obtained for the Epi-Rez 5101 and nylon system with the nickel adherend because this system would not adhere to the nickel and fell apart during handling of the specimens.

Drum-peel testing: The drum-peel test specimen shown in figure 8 was fabricated by applying the fabric-reinforced adhesive to the outer surface of a metal ring (mandrel). To represent each liner material, the metal rings were fabricated from either 6061-T6 bare aluminum or nickel-plated mild steel. Two plies of the preimpregnated tape were wrapped onto the surface of the adhesive-coated mandrel with 40 lb of tension on the tape. Just before the tape-wrapping operation, a 2 in. x 1/2 in. strip of 1-mil teflon was applied to the surface of the mandrel. After cure, a cut was made into the epoxy-glass composite to the edge of the teflon strip. The section of tape above the teflon strip was peeled away from the mandrel. This created a tab which could be gripped in the jaws of a testing machine.

Detailed specimen processing is described in Appendix A.

Five specimens each of the six candidate adhesive systems for each liner were tested at +75°F, -320°F, and -423°F. Results are shown in figures 9 and 10.

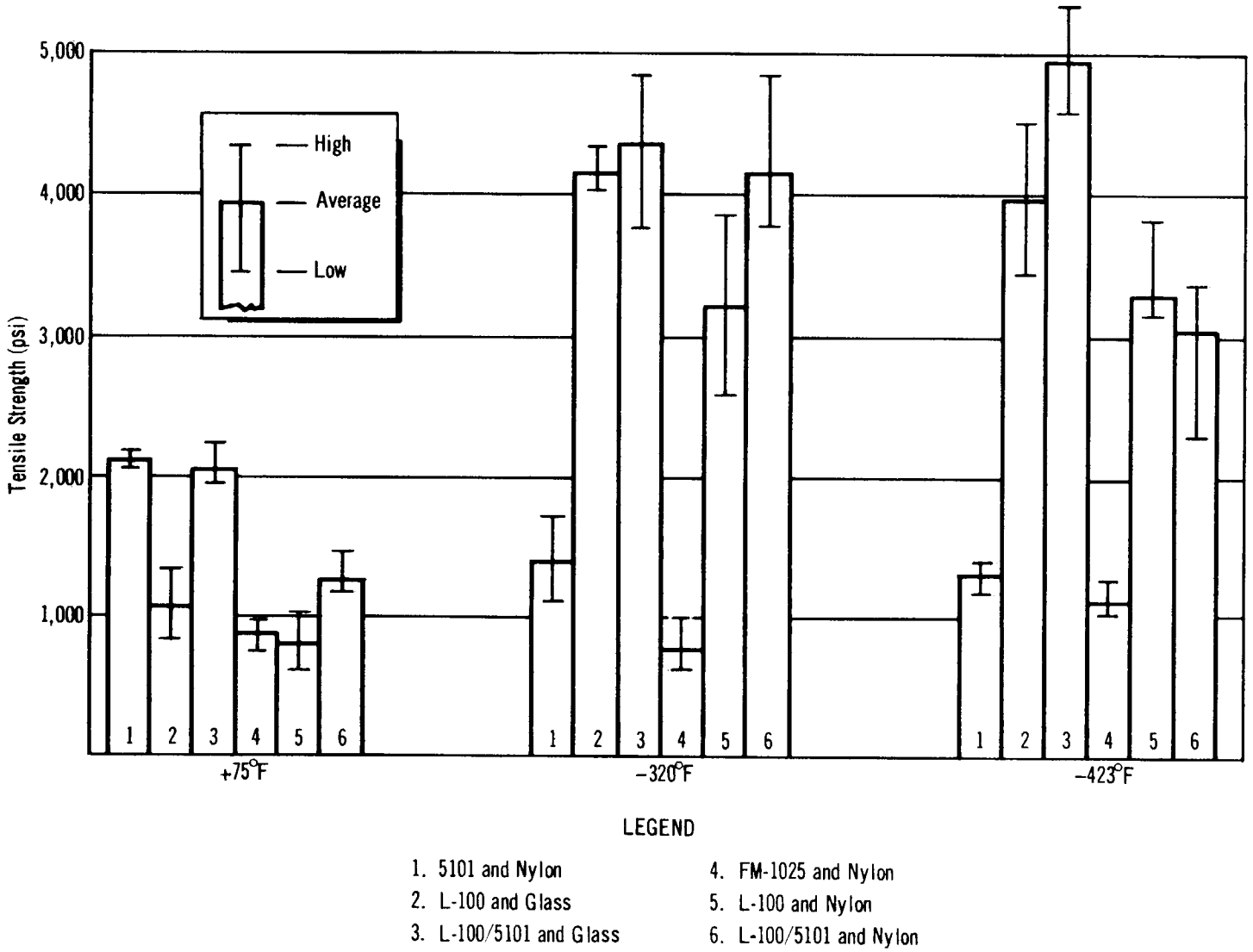


Figure 6. Tensile Lap Shear Tests, Aluminum Adherends

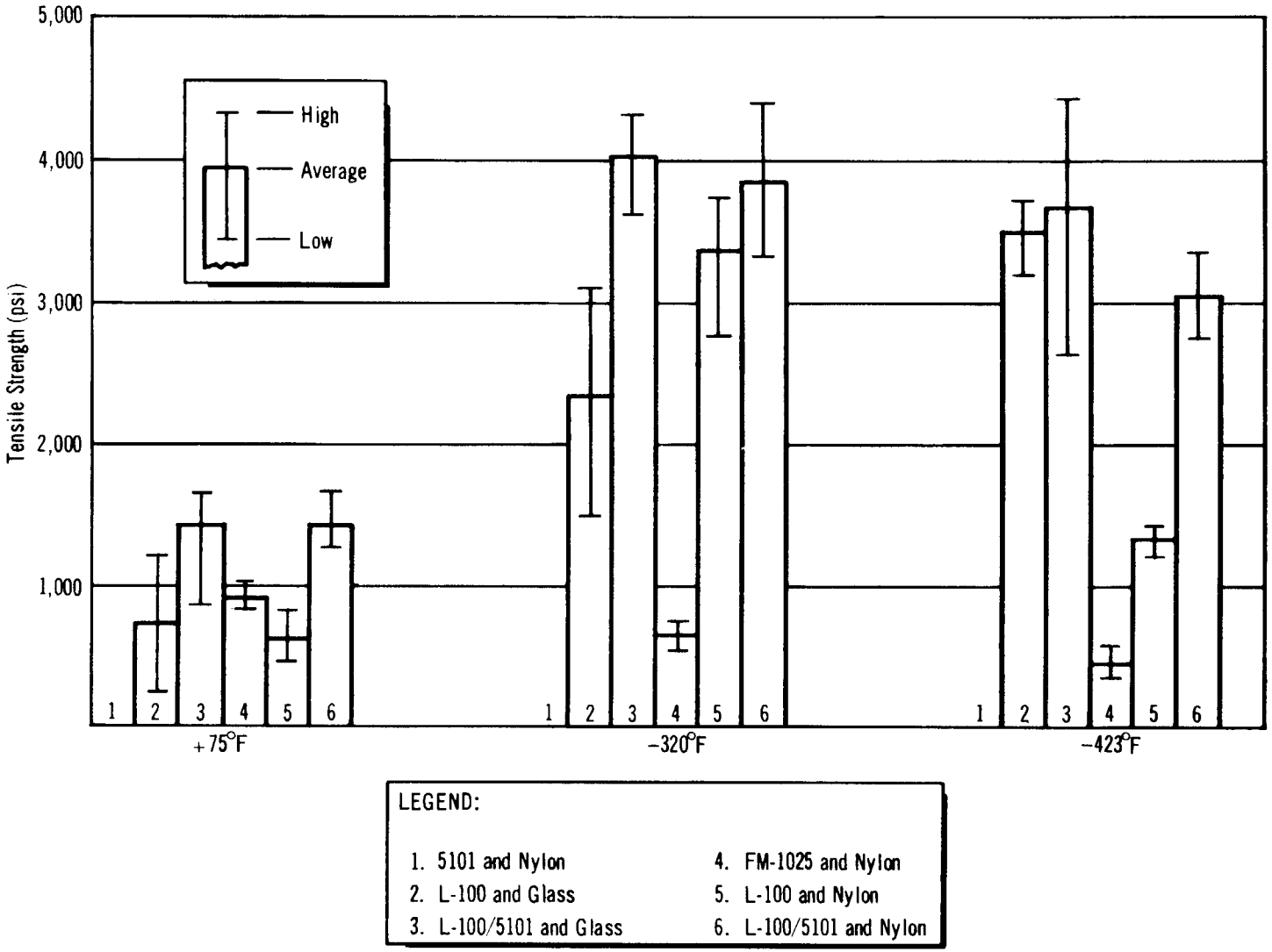


Figure 7. Tensile Lap Shear Tests, Nickel Adherends

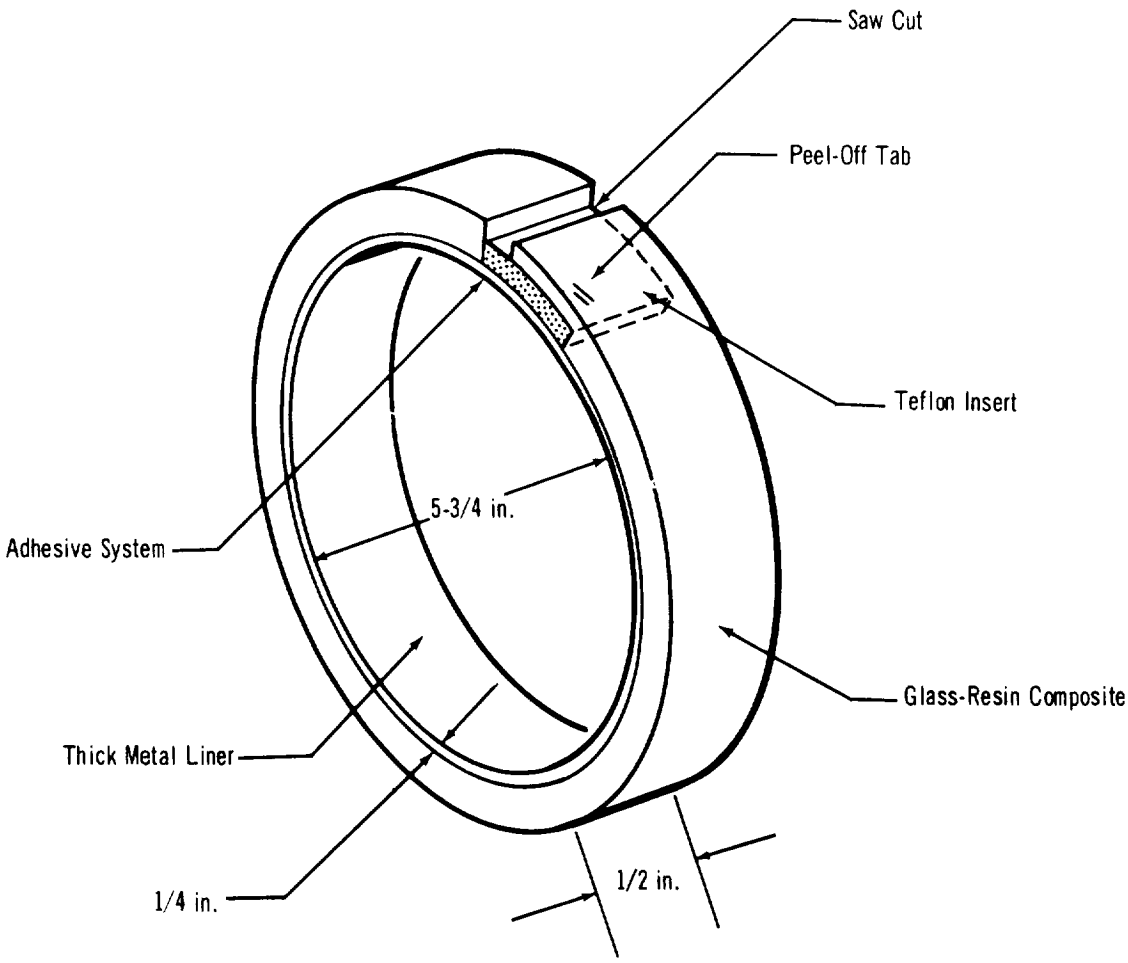


Figure 8. Drum-Peel Test Specimen

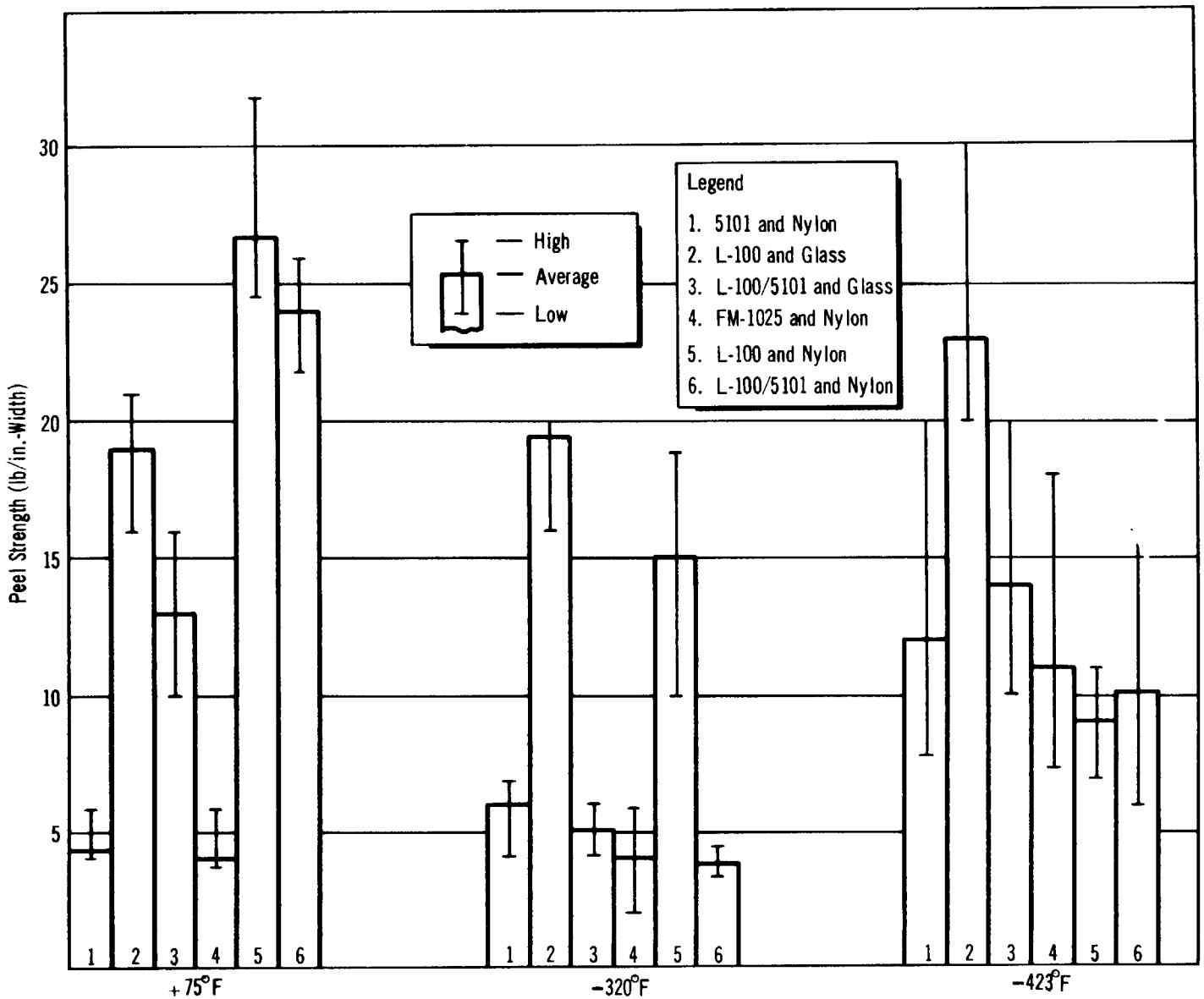
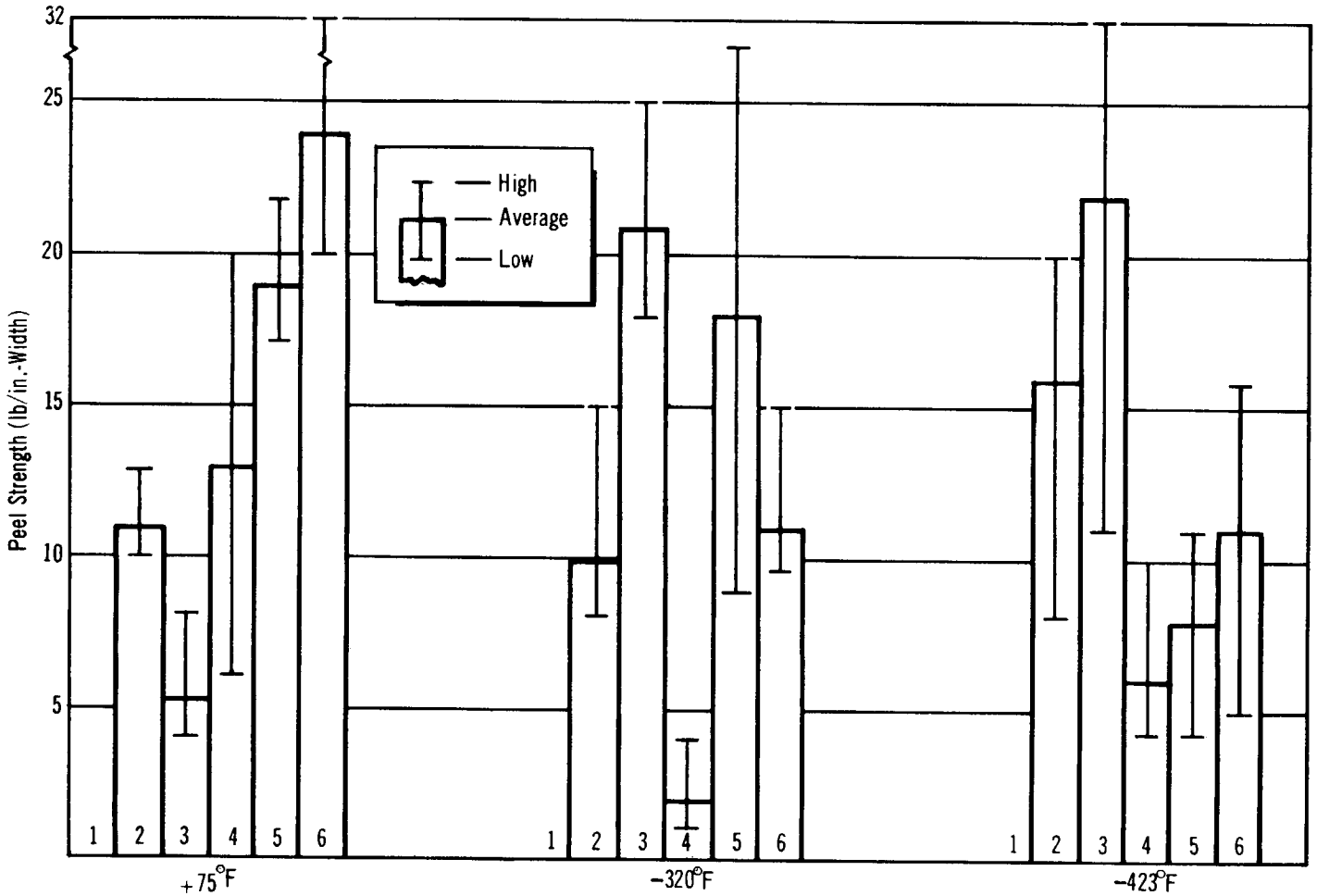


Figure 9. Drum Peel Tests – Aluminum Base



Legend:

1. 5101 and Nylon
2. L-100 and Glass
3. L-100/5101 and Glass
4. FM-1025 and Nylon
5. L-100 and Nylon
6. L-100/5101 and Nylon

Figure 10. Drum Peel Tests, Nickel Base

The testing jig consists of a simple drum and yoke mechanism (fig. 11). The metal-glass composite ring is inserted in the testing jig and the "peel-off tab" is gripped in the jaw of the testing machine. Once started, the specimen and drum continue to rotate until the glass filament section of the ring composite is peeled off the metal ring. During the test, load versus machine crosshead travel is recorded. After a constant force has been reached, the average peel strength in lb/in. of specimen width is determined. The rate of crosshead travel was held constant for all testing (6 in./min).

For tests at cryogenic temperatures, the specimens were soaked for a suitable period of time until thermal equilibrium was reached (minimum specified time of 10 min.).

From the data shown in figures 9 and 10, the following conclusions can be drawn. The polyurethane systems exhibited higher peel strengths with aluminum than the epoxy systems, except at -423°F. The polyurethane-epoxy blends exhibited peel strengths with aluminum close to the polyurethane systems, except at -320°F. With a nylon scrim, the polyurethane and polyurethane-epoxy blend showed a decrease in peel strength with nickel at cryogenic temperatures. With a glass scrim, the polyurethane and polyurethane-epoxy blend showed an increase in peel strength with nickel at cryogenic temperatures. The average peel strengths of all adhesive systems with nickel were about equal to the average peel strengths with aluminum.

Preliminary adhesive selection: From the lap-shear and drum-peel work, the 70:30 blend of polyurethane/epoxy/glass scrim showed the best shear strength with the aluminum adherends, except at -320°F. The L-100/glass, L-100/nylon, and L-100/5101/nylon also showed good shear strengths. Adhesive system L-100/glass had the best overall peel strength. The L-100/glass, L-100/5101/glass, and L-100/nylon were selected for further investigation for use with aluminum liners.

For nickel, the 70:30 blend of polyurethane/epoxy/glass (L-100/5101/glass) showed the best shear strength at all temperatures and the highest peel strength at cryogenic temperatures. The L-100/5101/nylon ranked as one of the best three systems in all of the tests. Adhesive system L-100/nylon ranked higher for the higher temperatures than the L-100/glass. The L-100/5101/glass, L-100/nylon, and L-100/5101/nylon were selected for further investigation for use with nickel liners.

Therefore, for the two metallic liners, a total of four adhesive systems was selected for further investigation.

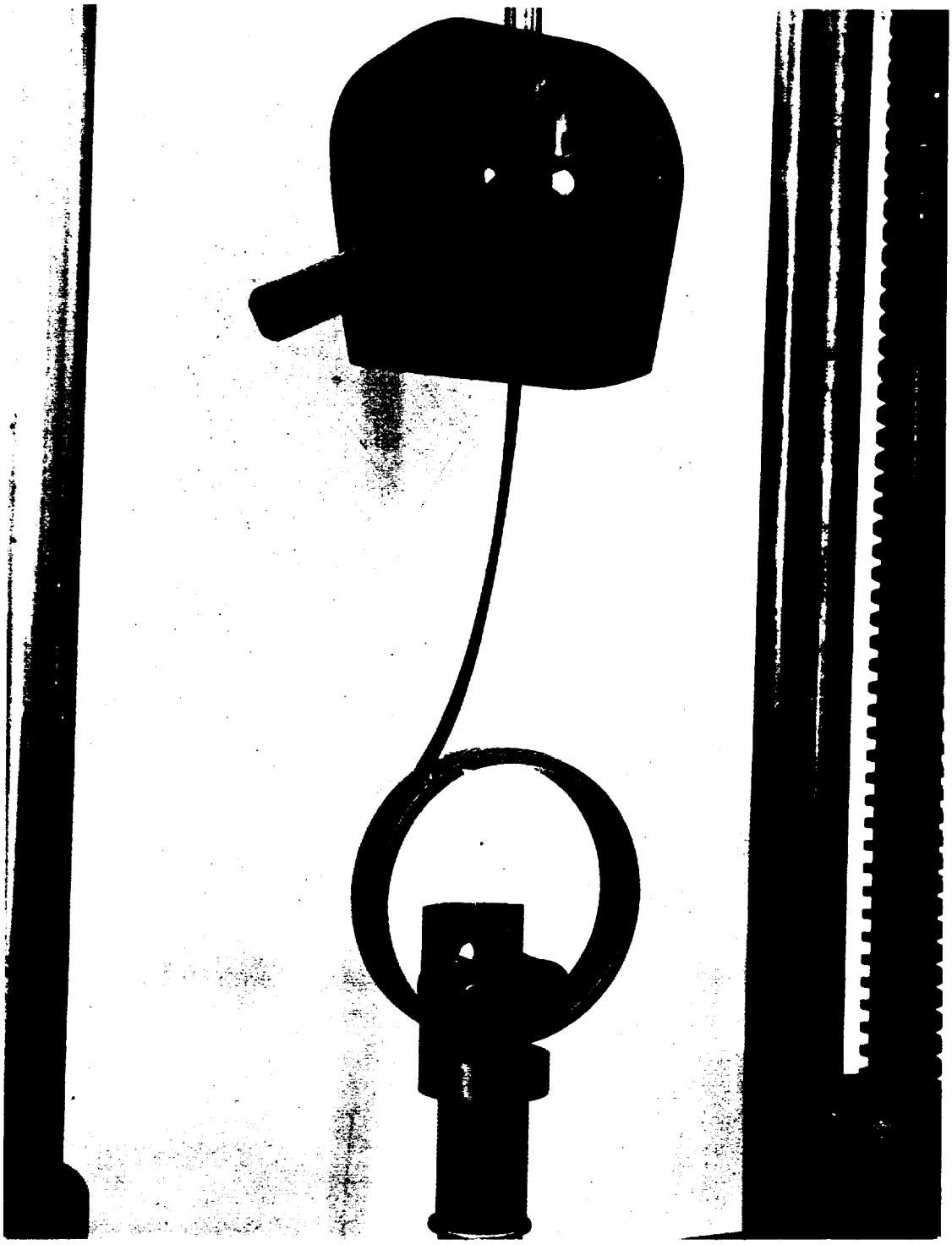


Figure 11 Drum Peel Test Setup

Uniaxial tensile testing: To determine the extensibility and tensile strength of the selected adhesive systems, the four adhesive systems were evaluated in uniaxial tensile tests at +75°F, -320°F and -423°F. The test results are shown in table II.

To effectively utilize the limited material, the tensile test specimen conformed to the standard die C dumbbell shape given in ASTM tentative standard D412-62T for vulcanized rubber. The test specimens were cut from a cured cast laminate of each adhesive system (fig. 12).

Detailed specimen processing is described in Appendix A.

TABLE II

ADHESIVE SYSTEMS MECHANICAL PROPERTIES DATA

	Adhesive systems	Test temp. °F	Average[a] ultimate tensile, psi	Average percent elongation	Average[a] elastic modulus, psi
1.	Adiprene L-100 and MOCA with nylon reinforcement	+ 75	6 950	37.0	19 000
		-320	14 000	8.3	547 000
		-423	11 730	3.8	562 700
2	Adiprene L-100 and MOCA with glass reinforcement	+ 75	5 380	5.3	162 500
		-320	14 100	4.8	696 000
		-423	11 730	2.4	715 100
3.	Adiprene L-100 Epi-Rez 5101 (70:30) and MOCA with glass reinforcement	+ 75	9 070	4.2	369 000
		-320	35 600	4.3	896 700
		-423	33 730	3.9	1 072 000
4.	Adiprene L-100 Epi-Rez 5101 (70:30) and MOCA with nylon reinforcement	+ 75	9 600	32.0	68 270
		-320	15 070	13.0	293 000
		-423	11 780	7.8	356 000

[a]Average of three test specimens.

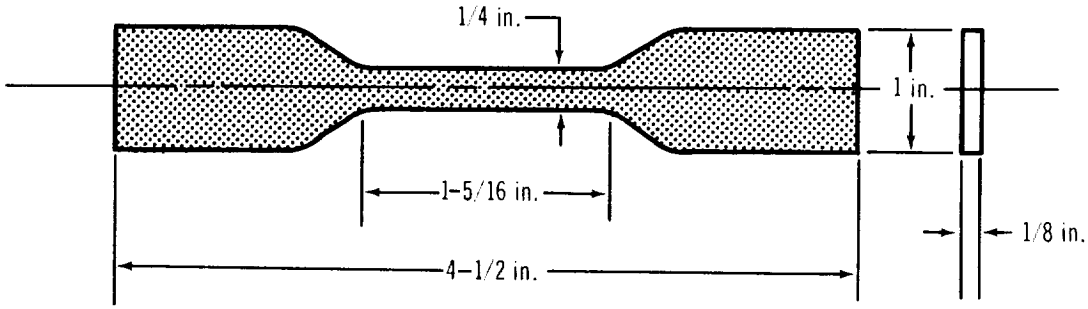


Figure 12. Uniaxial Tensile Specimen

Three specimens of each selected adhesive system were tested at each of the three temperatures in accordance with ASTM tentative standard D638-61T for testing plastics. For tests at cryogenic temperatures, the specimens were soaked until thermal equilibrium was reached (minimum specified time of 10 min.). The specimens were tested at a head travel rate of 0.1 in./min.

From the data shown in table II the following conclusions can be drawn. The adhesive systems with a nylon scrim exhibited higher ultimate elongations than the adhesive systems with a glass scrim. For a given scrim cloth, the L-100/5101 blend adhesive exhibited higher elongations at cryogenic temperatures than the pure L-100 adhesive.

Coefficient of contraction testing: To determine if the contraction of the adhesive system was compatible with that of the liner and glass-fiber composite, the four adhesive systems were evaluated in thermal contraction tests from +75°F to -103°F, -320°F, and -423°F. The data are shown in figure 13.

Tests were performed in accordance with ASTM specification E228-63T. The test length of each specimen was measured at room temperature, and the specimen was then cleaned and installed in a dilatometer. The contraction measurements were performed by cooling the dilatometer between any two temperatures and leaving the dilatometer at each temperature until the extensometer showed no significant change. Two specimens of each adhesive system were tested.

From the data shown in figure 13, it can be concluded that the scrim had a greater effect on the thermal contraction of the adhesive systems than changes in the adhesive resin.

Preliminary Vessel Adhesive Selection. - Table III presents a summary of the uniaxial tensile and thermal contraction data.

TABLE III

ADHESIVE COMPARISON

Mechanical property	Adhesive system			
	L-100/ glass	L-100/5101/ glass	L-100/ nylon	L-100/5101/ nylon
Ultimate uniaxial elongation of adhesive system, % at -423°F.	2.4	3.9	3.7	7.8
Chilldown differential, % (difference in contraction between adhesive system and fiberglass composite, +75° to -423°F.)	0.4	0.4	0.8	1.0
Residual uniaxial elongation of adhesive system, % at -423°F.	2.0	3.5	3.1	6.8

The design working strain of the Task II pressure vessel was to be 2% biaxially. Therefore, it was mandatory that the residual uniaxial elongation of the selected adhesive be at least 2%. The adhesive systems L-100/5101/ glass and L-100/nylon both exhibited available elongations greater than the vessel design strain. Together with the tensile-lap-shear and drum-peel tests, this indicated the selection of the L-100/5101/glass system as the most promising for use with both aluminum and nickel liners.

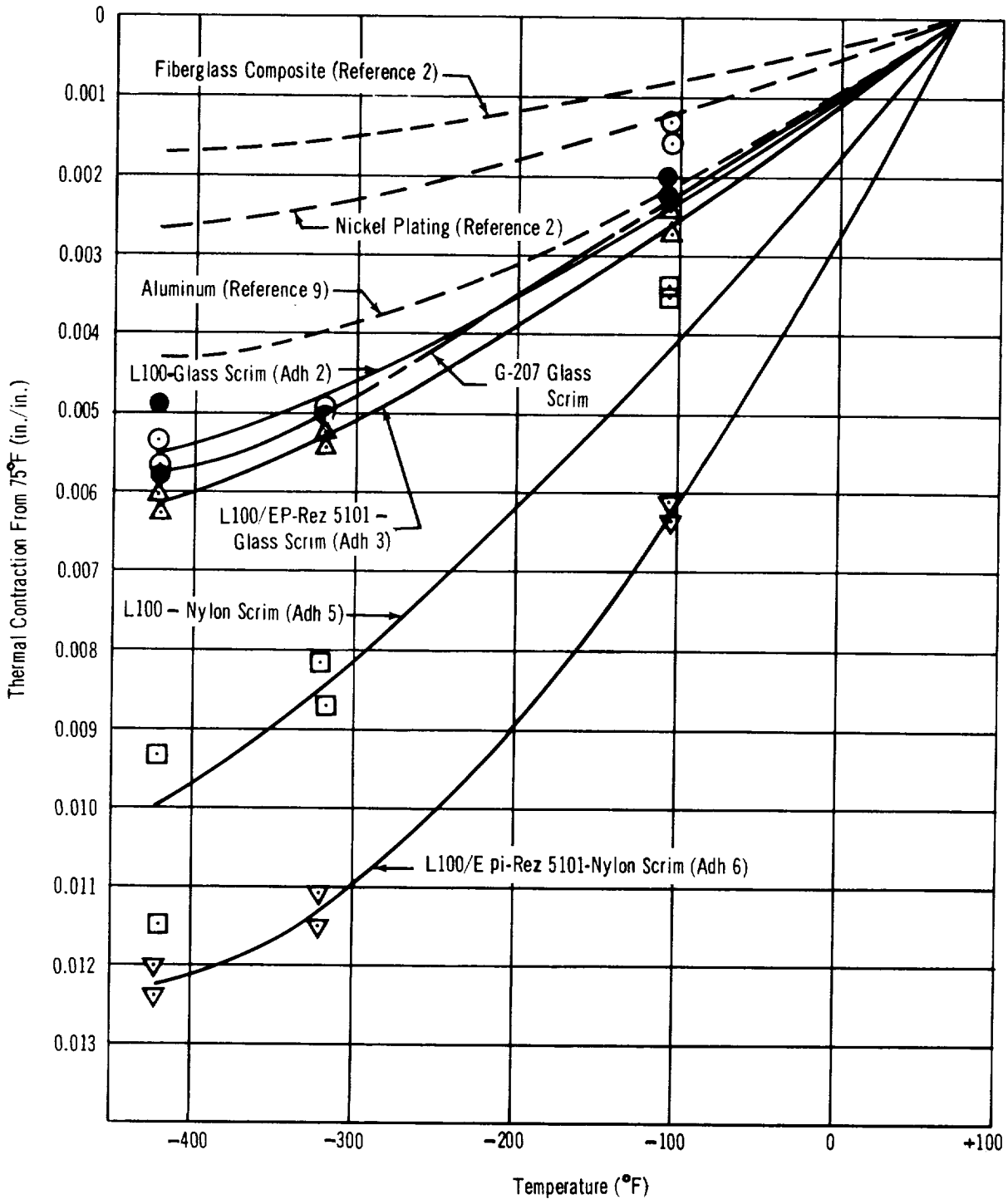


Figure 13. Contraction Curves of Selected Adhesives and Other Vessel Components

Additional Adhesive Evaluation. - Additional work was conducted throughout the remainder of the program based upon the potential established by research at Lewis Research Center and the Task III pressure vessel testing.

An experimental adhesive was selected for evaluation based upon Lewis Research Center work. The adhesive is a linear, saturated, thermoplastic, polyester, resin system manufactured by Goodyear Aerospace Corporation and designated Goodyear G-207. The adhesive with the No. 112 glass scrim cloth with A-1100 silane finish was evaluated by the same test methods previously described except that no work was done with nickel components.

Polyester G-207: Results of the lap-shear testing are shown in table IV. Comparing these with the values of the other adhesives given in figure 6, the G-207 lap-shear strengths, both with and without scrim cloth, are considerably below most of the other adhesives.

TABLE IV

TENSILE SHEAR TEST RESULTS FOR GOODYEAR G-207 ADHESIVE WITH ALUMINUM AND EPOXY-GLASS COMPOSITE ADHERENDS

Scrim cloth (with or without)	Test temp, °F	Average shear strength,[a]
Without	+77 ± 5	454
With	+77 ± 5	182
Without	-320	1255
With	-320	938
Without	-423	1222
With	-423	485

[a] Average of five specimens

Results of the drum-peel testing are shown in table V. Comparison of these with the values of the other adhesives given in figure 9 shows the drum-peel strength of G-207 without scrim to be comparable to the best adhesive at -320°F and -423°F and considerably lower than most at +75°F. The drum-peel strength of the G-207 with scrim is only slightly higher at +75°F and considerably lower at -320°F and -423°F than the G-207 without scrim.

TABLE V

DRUM-PEEL TEST RESULTS FOR G-207 ADHESIVE
WITH ALUMINUM AND EPOXY-GLASS COMPOSITE ADHERENDS

Scrim cloth (with or without)	Test temp, °F	Average shear strength, lb/in. width
With	+75	10[a]
Without	+75	2[b]
Without	+75	9[a]
With	-320	15[c]
Without	-320	24[c]
Without[d]	-320	12[b]
With[d]	-320	11[e]
With[c,b]	-423	18[a]
Without	-423	22[a]

[a]Average of four specimens.
[b]Average of three specimens.
[c]Average of five specimens.
[d]Unprimed aluminum surface.
[e]One specimen.

Considerable difficulty was encountered in the fabrication of specimens because of the high volatile content (72%) of the basic adhesive-resin system. It was not possible to make a cast specimen of the basic adhesive-resin. A limited number of scrim cloth specimens were made by continually immersing and drying 12 sheets of No. 112 glass cloth with the adhesive and finally curing the resulting impregnated plies for 2 hr at 300°F and under a pressure of 1000 psi (details of specimen fabrication are given in Appendix A).

Two tensile specimens were tested at each of the temperatures, +75°F and -320°F. The results are shown in table VI. The percent elongation is adequate at +75°F and satisfactory at -320°F. Insufficient material was available to fabricate specimens for testing at -423°F.

The thermal contraction characteristics of the material (specimen processing is given in Appendix A) are shown in figure 13. The data are comparable to the characteristics of the L-100/MOCA glass cloth system and the L-100:Epi-Rez 5101 (70:30 pbw)/MOCA glass cloth system, which indicated again that the thermal contraction behavior was influenced primarily by the scrim rather than the matrix.

Other additional adhesive work: Results of the concurrent vessel testing in Task III indicated that greater ambient temperature tenacity of the adhesives was needed. As a result, five additional adhesive systems were evaluated by drum-peel tests, which represented the apparent severest single criterion for a successful application. The five systems evaluated were G-207 without a scrim cloth, G-207 with a G-207 impregnated nylon scrim cloth, G-207 with an Epi-Rez 5101/APCo 322 impregnated nylon scrim cloth, L-100:Epi-Rez 5101(70:30)/MOCA with a nylon scrim cloth, and L-100:Epi-Rez 5101(80:20)/MOCA with a nylon scrim cloth.

The nylon scrim cloth* which was evaluated here was thinner and had a tighter weave than the previously used nylon scrim cloth.

It was desirable that the cure of the two blended systems and the winding resin be definitely established. This was accomplished through the use of the vibrating-reed test method.

The vibrating-reed test method is based on the principle that a reed-shaped specimen when subjected to forced transverse vibration has a resonant frequency dependent upon its physical parameters and Young's modulus. During a curing polymeric reaction, the Young's modulus of the material increases as the molecular weight increases and the modulus approaches a constant value as the reaction nears completion. The vibrating reed apparatus follows the cure cycle of a material by measurement of the changes in the amplitude of the frequency response as a function of time. From this information, the modulus (cure) may be determined.

*Nylon scrim cloth No. 34168-2 (scoured and heat-set) supplied by J. P. Stevens and Company.

TABLE VI

UNIAXIAL TENSILE TEST RESULTS FOR G-207 ADHESIVE

Specimen No.	Test Temp, °F	Width, in.	Thickness, in.	Area, in^2	Load, lb	Tensile strength, psi	Elongation, %	Modulus, psi
1	+75	0.239	0.0858	0.0205	118	5 785	10.3	74 000
2	+75	0.266	0.0950	0.0215	112	5 210	7.7	92 900
3	-320	0.206	0.0988	0.0204	317	15 540	2.7	383 000
4	-320	0.246	0.0960	0.0233	540	23 175	4.9	307 000

Details of the test procedure and detailed results are given in Appendix B. In summary, the testing indicated that the cure for each system was essentially complete in approximately 9 hr, 8-1/2 hr, and 9-1/2 hr for the Epi-Rez 5101/APCo 322, L-100:Epi-Rez 5101(80:20)/MOCA, and L-100: Epi-Rez 5101(70:30)/MOCA, respectively.

The drum-peel test specimens were fabricated similarly as that used previously. The cure cycle was changed to reflect the vibrating-reed work. Details are given in Appendix A.

The drum-peel test results are shown in figure 14, which is a replot of figure 9, with the addition of the new data.

The 80:20 L-100/5101 system was vastly superior in peel-strength to any of the adhesives tested. The peel-strength of the 70:30 system was slightly better with the new nylon compared to the previous nylon. The peel-strength of the G-207 without scrim was average, while the G-207 with glass scrim and nylon scrim was below average.

With the advice and approval of the NASA LeRC Center Program Manager, the 80:20 and the 70:30 L-100:5101/nylon scrim systems, and the G-207 without scrim system were selected as offering the most potential for satisfactorily bonding the thin metal liner to the composite wall. These systems were evaluated in vessels during the latter phase of the program.

Task II - Pressure Vessel-Liner Development and Fabrication

Concurrently with Task I, an open-ended subscale pressure vessel was designed for evaluation of the candidate liners' ability to withstand repeated cyclic strains to 2% when bonded to the vessel wall. The longitudinal-to-circumferential strain ratio in the test section was designed for 1/1 at as low an internal pressure as possible.

Points considered were the following:

(1) Configuration.

(2) Design.

(3) Liner materials.

(4) Fabrication.

(5) Verification and development of design.

(6) Test-vessel fabrication.

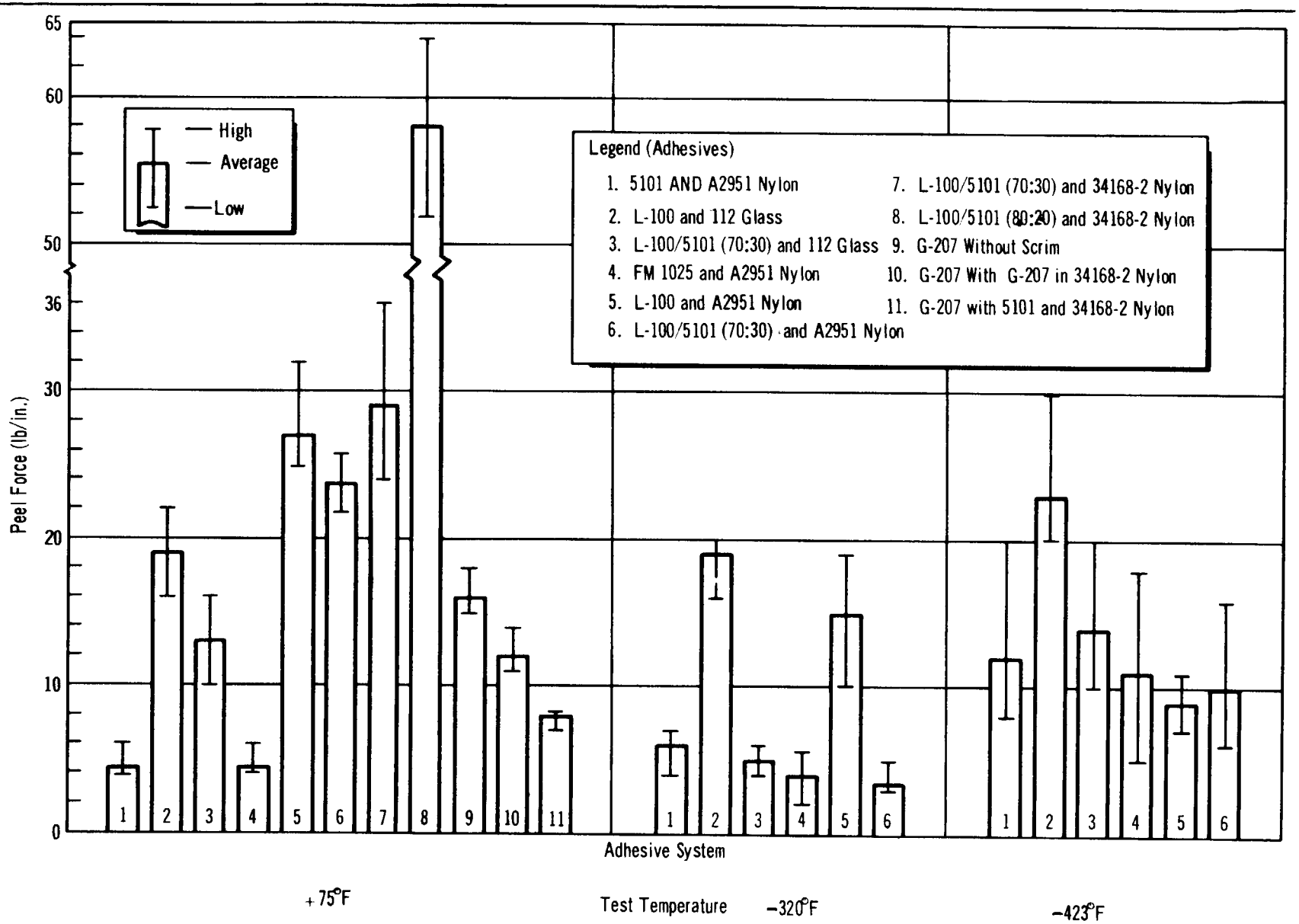


Figure 14. Drum Peel Test Results – Aluminum Base

Configuration. - A 7-1/2-in.-diam by 20-in.-long, open-ended cylinder vessel configuration was chosen for the program (fig. 15). This same configuration had been used for numerous Douglas Independent Research and Development programs and for the liner screening of Contract No. NAS 3-2562 (ref. 2). The configuration was simple; compound curvature effects were eliminated, and the vessel interior was easily accessible for liner examination and strain gage application. The flanged design is also suitable for standard cryogenic sealing techniques.

The vessels fabricated on Contract No. NAS 3-2562 utilized longitudinal No. 9943 woven glass cloth (S994/HTS), single-end roving as circumferential reinforcement (S994/HTS), and Nos. 181 and 1584 (E/Volan A) woven glass cloth in the build-up areas. However, to fully simulate a filament-wound structure and to achieve greater composite uniformity, vessels for this program were fabricated with Douglas-developed, preimpregnated, collimated, glass-fiber tape.

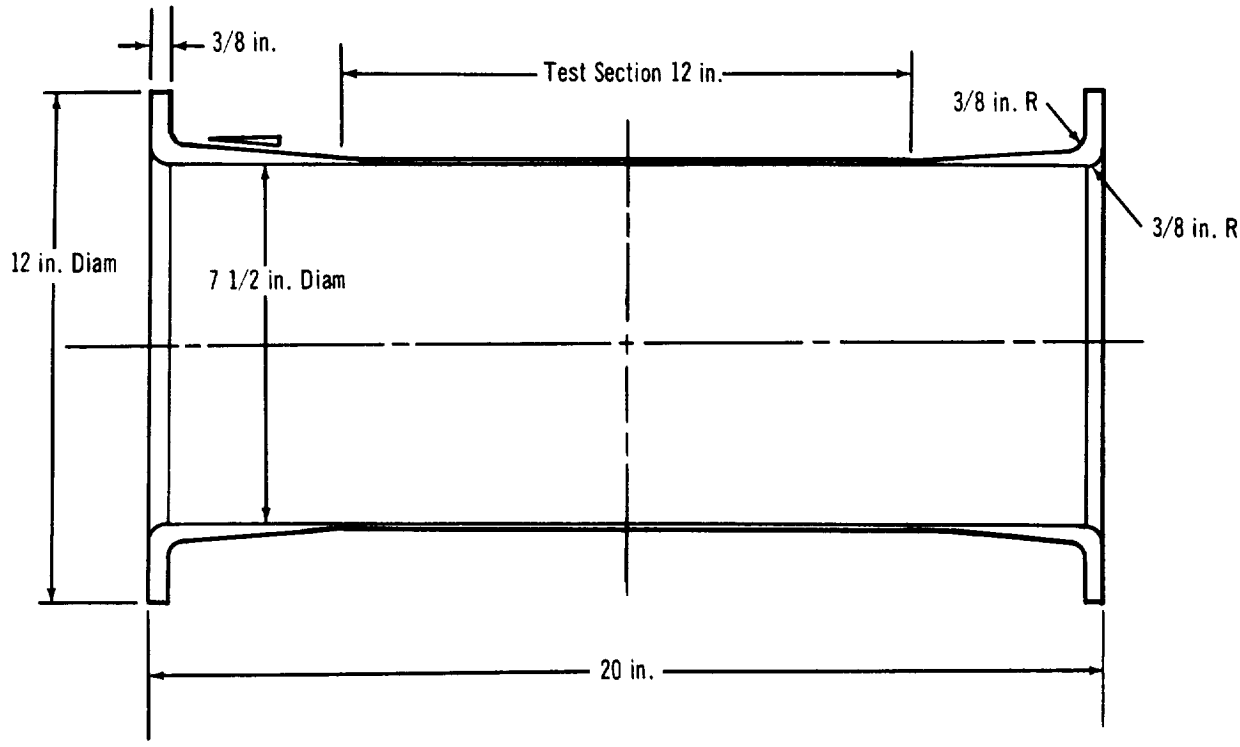


Figure 15. Subscale Pressure Vessel

Design. - The vessel was designed by netting analysis to achieve a longitudinal-to-circumferential strain ratio of 1/1 in the test section. This was accomplished by using one longitudinal layer and two hoop layers of the preimpregnated collimated glass-fiber tape. The design was approved by the NASA LeRC Project Manager.

Liner Materials. - Electrodeposited nickel and aluminum foil were the two liner materials selected for the program.

Aluminum: Aluminum foil type A1100-0 of 2-mil thickness was selected as a liner material for the cylindrical section of the pressure vessel. The end flanges of the aluminum-lined vessels were fabricated with annealed, 6-mil aluminum (A1100) foil.

Electrodeposited nickel: Numerous vendors were solicited to bid on the liner electrodeposition and five vendors responded. Based on the evaluation of the bids received, one vendor was selected to do the work. However, during the preparation of the subcontract, the vendor notified Douglas that because of a recent overloading of personnel, he could not accept the subcontract.

Further discussion with one of the bidders, Lockheed Missiles and Space Company, led to the award of a development subcontract.

Cyclic Behavior: - During cyclic pressurization of a metallic-lined fiberglass vessel to 2.0% strain, the following sequence of events is believed to occur (fig. 16).

(1) All components strain elastically to point (1) shown in figure 16.

(2) The metallic liner strains plastically to (2). Glass fiber composite is assumed elastic to (2).

(3) Upon release of pressure, the liner returns elastically to zero load (3). The fiberglass load-strain relationship is assumed to be the same for both loading and unloading.

(4) The metallic liner is compressed to (4), its compressive-yield stress. This is probably lower than the tensile-yield stress because of the Bauschinger effect.

(5) The liner is strained plastically in compression until the tensile load in the fiberglass composite is matched by the compressive load in the metallic liner at (5). The internal pressure of the vessel is now zero, and it retains a residual strain ε_r.

(6) Upon repressurization of the vessel, the compressive load in the liner is relieved so that it strains elastically to point (6); the load in the liner is now zero. The liner continues to strain elastically to point (7). The yield point of the liner on the second cycle is unknown; it may be the same or higher or lower than the yield point on cycle one.

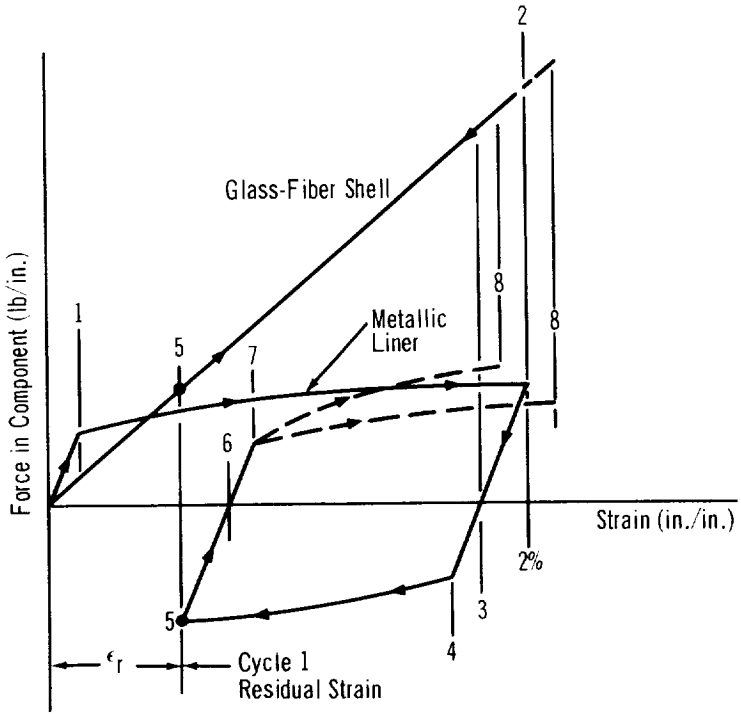


Figure 16 Probable Composite Cyclic Behavior

(7) The liner is again strained plastically in tension. Because the vessel is cyclic pressurized to the same internal pressure, the total force in the structural wall (liner plus glass-fiber composite) must remain the same. If subjected to strain hardening, the liner will accept more load than it did on cycle No. 1. Consequently, there is less load for the glass-fiber shell to accept, and the strain achieved by the vessel will be less on cycle No. 2 (Point 8). Conversely, subjected to strain softening, the liner will accept less load on cycle No. 2. The glass-fiber shell is forced to accept more load, and the strain achieved by the vessel will increase on cycle No. 2 (Point 8').

The number of cycles that can be achieved by a fiberglass vessel operating at a strain of 2% depends on the bonded liner's resistance to fracture when subjected to high plastic-tensile and compressive strains associated with pressure cycles as illustrated in figure 16.

To fully evaluate the actual load-strain relationship of the fiberglass shell and metal liner, it would be advantageous to have the stress-strain diagram of the metallic liner for each cycle. It would then be possible to determine the load taken by the liner during any selected pressure cycle.

Such an evaluation was undertaken with aluminum; original work with nickel was discontinued when the final estimated cost of specimen fabrication turned out to be more than an order of magnitude higher than the initial estimated cost.

Specimen configuration was as shown in figure 17. Two specimens were to be tested at ambient temperature; two were to be tested at -320°F. Ambient temperature testing was started first.

During the tensile loading to 2% strain, the soft aluminum specimens necked down excessively. On subsequent compressive cycles, bending occurred in the specimens. The localized strain in the necked down section was greater than the average 2% strain over the 1-in. gage length, because of both necking down and bending. Therefore, quantitative results for the ambient temperature tests were lacking. However, qualitatively, the A1100 aluminum strain-softened asymptotically during the elasto-plastic deformations. There was an approximate 20% reduction in load-carrying capacity of the material at the end of 100 cycles.

The liquid nitrogen testing was not attempted because of the inconclusive ambient temperature results.

Because of the difficulties involved in obtaining quantitative test results and because the strength contribution of the aluminum liner to the overall filament-wound composite structural shell was very small (less than 1%), the work was discontinued with the advice and approval of the LeRC Program Manager.

Vessel Fabrication. - A general description of the vessel fabrication follows. Detailed processing procedures are described in Appendix C. A summary of vessel fabrication is given in table VII.

Single-end yarn reinforcement: SCG 150 1/0 1.0 TPI "S" and "Z" twist yarns* with 901 finish (HTS) comprised the primary structural fiber reinforcement; it represented the most advanced product commercially available.

Glass-cloth reinforcement: E glass/Volan A Nos. 120, 181, and 1584 glass cloth was utilized for flange and build-up areas. This material has proven satisfactory in similar test specimens fabricated under Contract No. NAS 3-2562 (ref. 2).

Resin system: Epi-Rez 5101/APCo 322 resin system was used throughout the structural composite. Epi-Rez 5101 is a highly purified low-chlorine version of the standard resin, Epi-Rez 510. Manufacturing specifications call for a minimum of 0.1% total chlorine content. The epoxide equivalent weight is 185 - 200.

*Manufactured by Owens-Corning Fiberglas Corp.

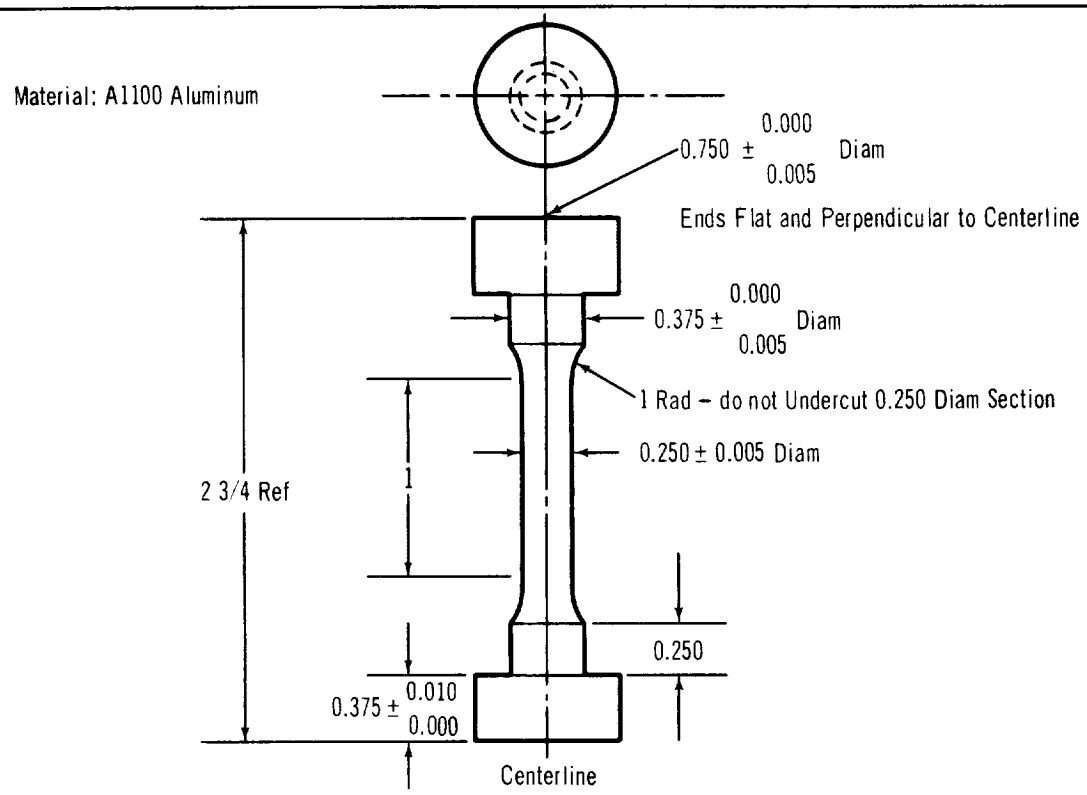


Figure 17. Tension Compression Specimen

APCo 322 is a high-heat distortion, aromatic, epoxy-resin hardener which produces excellent toughness. This hardener is a complex, highly functional, aromatic polyamine with no aliphatic side chains or aliphatic diamines present.

Douglas-preimpregnated, collimated, fiberglass tape: The resin system and fiber glass yarn reinforcement were combined in a special tape-making machine to produce Douglas-preimpregnated, collimated, fiberglass tape. This tape was used subsequently in the structural composite to form the cylindrical section of the subscale pressure vessel.

Douglas tape is a uniform collection of highly compacted and collimated, evenly tensioned, single-end yarns imbedded in a controlled "B-staged" resin matrix. It had been successfully used in several previous Douglas projects requiring reproducible parts having premium strength and closely controlled glass-resin composition. Preimpregnation allows close quality control of the tape, including resin content and processability, and collimation allows a high filament nesting in the final part.

Mandrel: The mandrel which was used for fabrication of the subscale pressure vessels is illustrated in figure 18. The mandrel is constructed of corrosion-resistant 321 stainless steel to provide durability. The essential parts are: a slightly tapered cylindrical section (for ease of removal) and two flanged sections. One flanged section is welded to the cylindrical portion. The other flanged section forms a very close, ridge-free fit with the cylindrical section when the drawbar assembly attached to the flanges is tightened.

A 10-in.-long cylindrical section is attached to one end of the mandrel and forms a tight fit at the mandrel end flange. This cylindrical section is identical to the mandrel in material, diameter, and surface finish. It was used to provide a quality control plate of the primary liner electroformed nickel. The 10-in. cylindrical section was removed for aluminum lined vessel fabrication.

Procedure: Electroformed liners were originally to be delivered in situ on the winding mandrel to the configuration shown in figure 19. Difficulties, which were encountered in electroforming to the tapered shape led to the incremental increase in thickness shown in figure 20. Aluminum liners were fabricated by wrapping one layer of foil onto the mandrel, which had been prepared with the end-flange aluminum liners already in place. The end-flange liners were fabricated to the required size and curvature in a female mold (fig. 21).

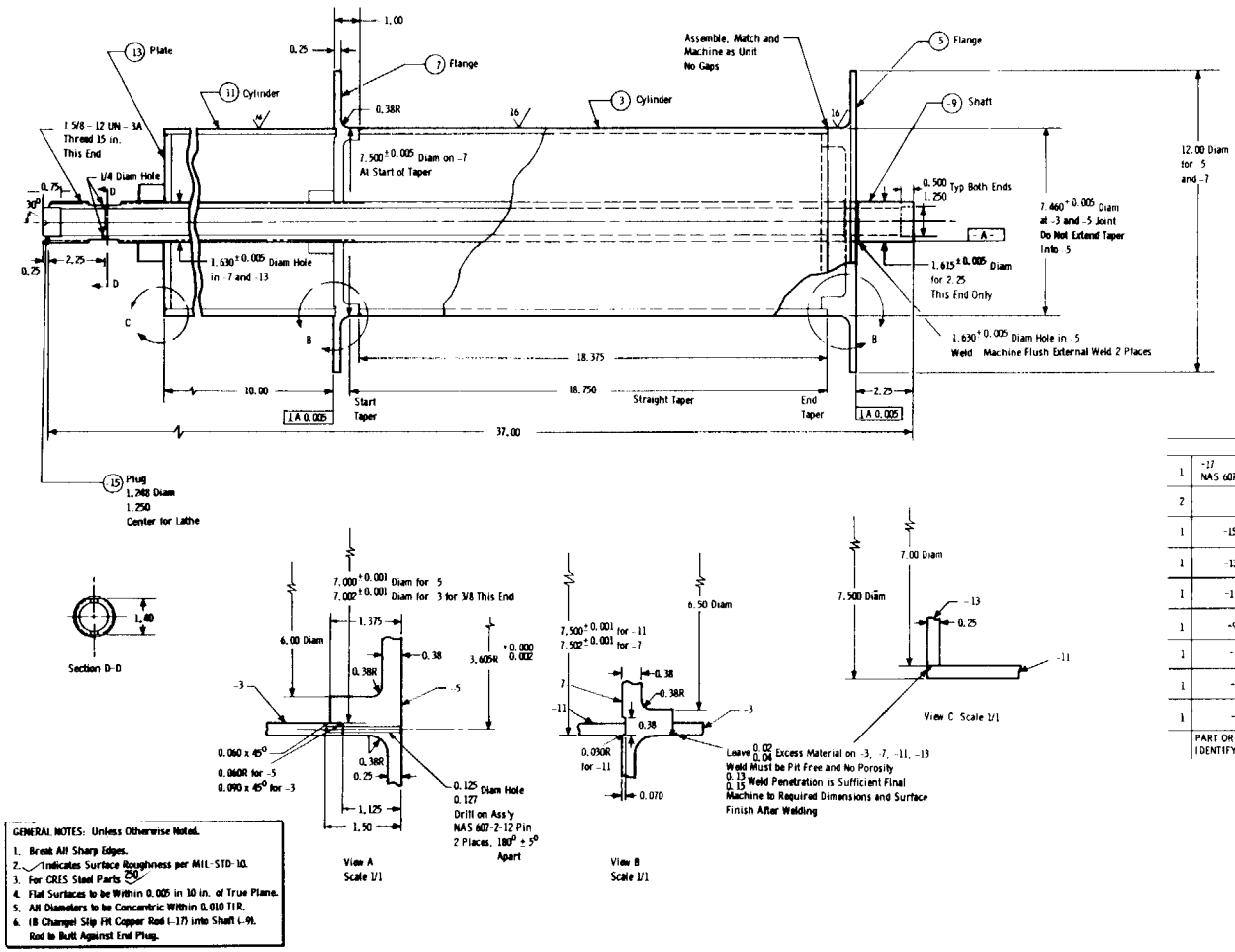
The liner surfaces were prepared for bonding as described in Appendix C.

The adhesive was then applied to the mandrel.

One hoop wrap of tape was wound over the adhesive to tie it down and apply pressure to increase the joint uniformity. The integral end-flanges were partially built up of preimpregnated Nos. 120, 181, and 1584 E/Volan A fiber glass cloth. The longitudinal tape was then laid down, the remainder of the end-flange fabricated, and a second hoop wrap applied over the cylindrical surface. Pressure rings were clamped on the end-flange build-up and clamps were used to tighten and pressure the laminate to assure a good quality, uniform thickness laminate. The part was then cured.

Process details are given in Appendix C. In table VII variations in fabrication are given for each of the vessels and are further described below.

Verification and Development of Design. - Following the initial design and NASA LeRC approval, two vessels were scheduled for fabrication and testing to verify the design strain field and to permit an evaluation and verification of the vessel end seals.



GENERAL NOTES: Unless Otherwise Noted.

1. Break All Sharp Edges.
2. ✓ Indicates Surface Roughness per MIL-STD-10.
3. For CRES Steel Parts ✓ 250
4. Flat Surfaces to be Within 0.005 in 10 in. of True Plane.
5. All Diameters to be Concentric Within 0.010 TIR.
6. (8 Change) Slip Fit Copper Rod (-17) into Shaft (-9). Rod to Butt Against End Plug.

LIST OF MATERIALS

	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	CODE IDENT NO.	STOCK SIZE	MATERIAL DESCRIPTION	MATERIAL SPECIFICATION	FIND NO.	ZONE
1	-17 NAS 607-2-12	INNER SHAFT		1 1/4 OD X37	COPPER ROD			
2		PIN						
1	-15	PLUG		3/4 x 1 1/2 x 1 1/2 GR. OPT	CRES PLATE TYPE 321	MIL-S-6721 COMP-Ti-ANLD		
1	-13	PLATE		1/4 x 8 x 8 GR. OPT	CRES PLATE TYPE 321	MIL-S-6721 COMP-Ti-ANLD		
1	-11	CYLINDER		8 OD x 1/2 x 10 1/2	CRES TUBE TYPE 321	AMS-5645		
1	-9	SHAFT		1 5/8 OD x1/4 x 37	CRES TUBE TYPE 321	A269W564		
1	-7	FLANGE		1 x 12 x 12 GR OPT	CRES PLATE TYPE 321	MIL-S-6721 COMP-Ti-ANLD		
1	-5	FLANGE		1 1/2 x 12 x 12 GR. OPT	CRES PLATE TYPE 321	MIL-S-6721 COMP-Ti-ANLD		
1	-3	CYLINDER		8 OD x 1/2 x 19	CRES TUBE TYPE 321	AMS-5645		

Figure 18. Mandrel for Cryogenic Test Pressure Vessel

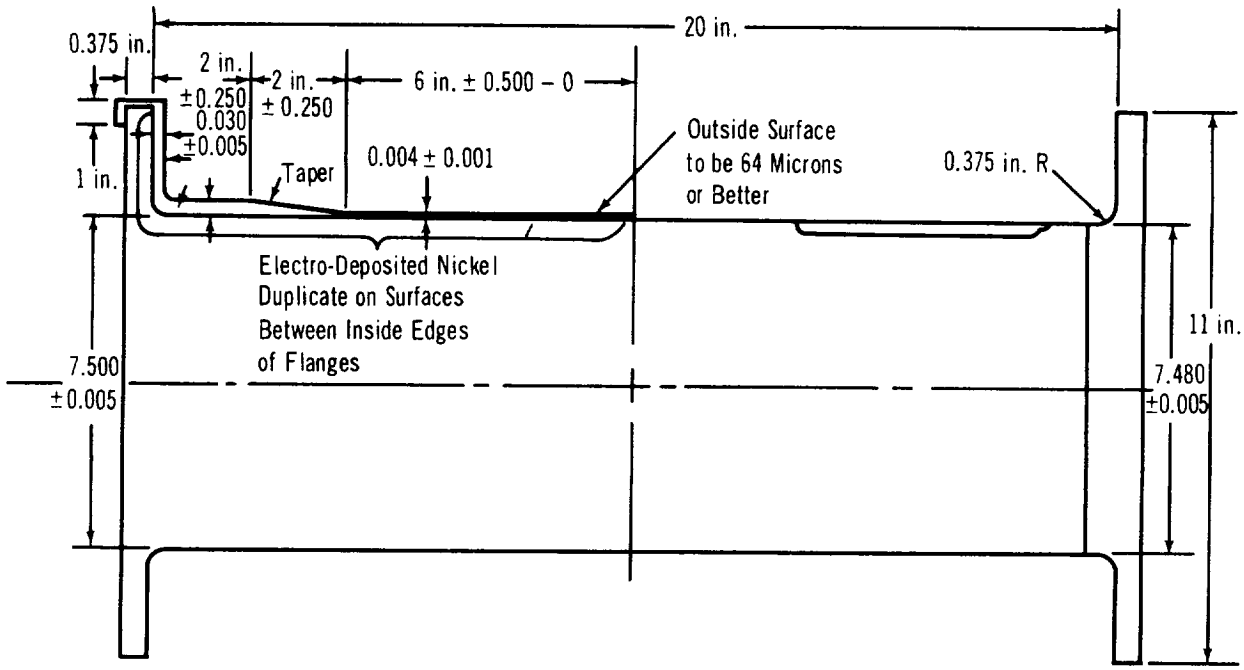


Figure 19 Electro-Deposited Nickel Liner

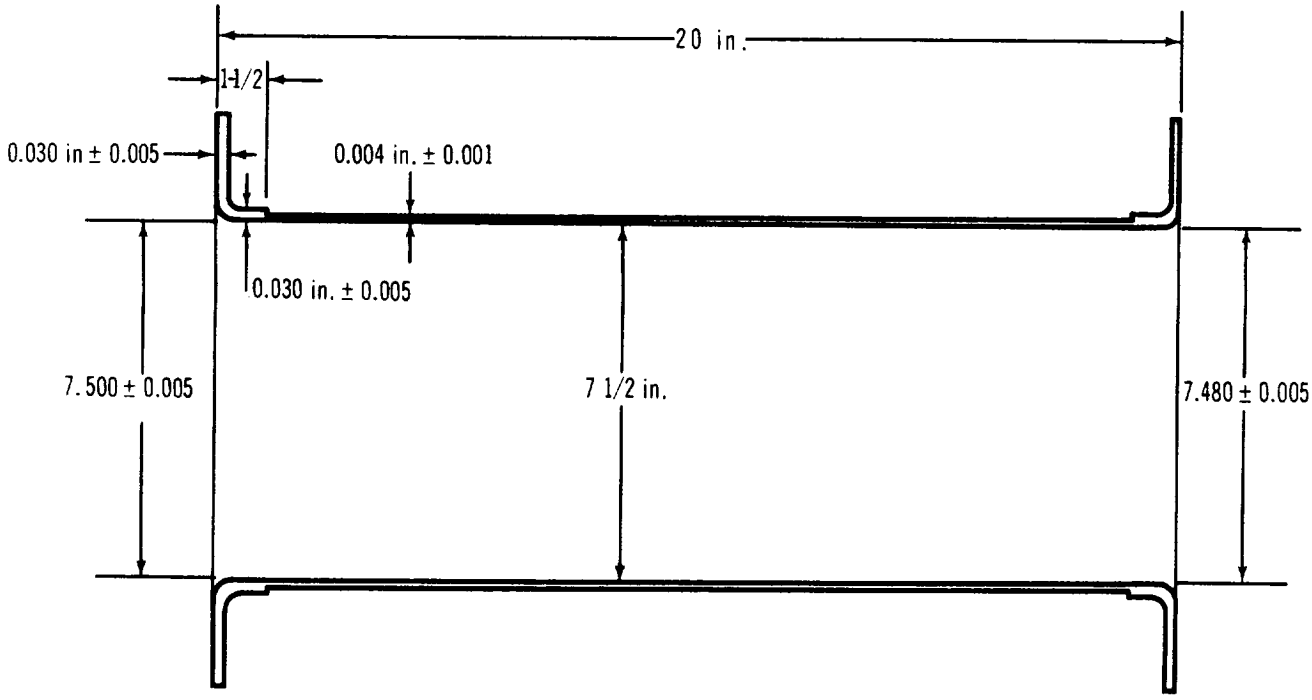
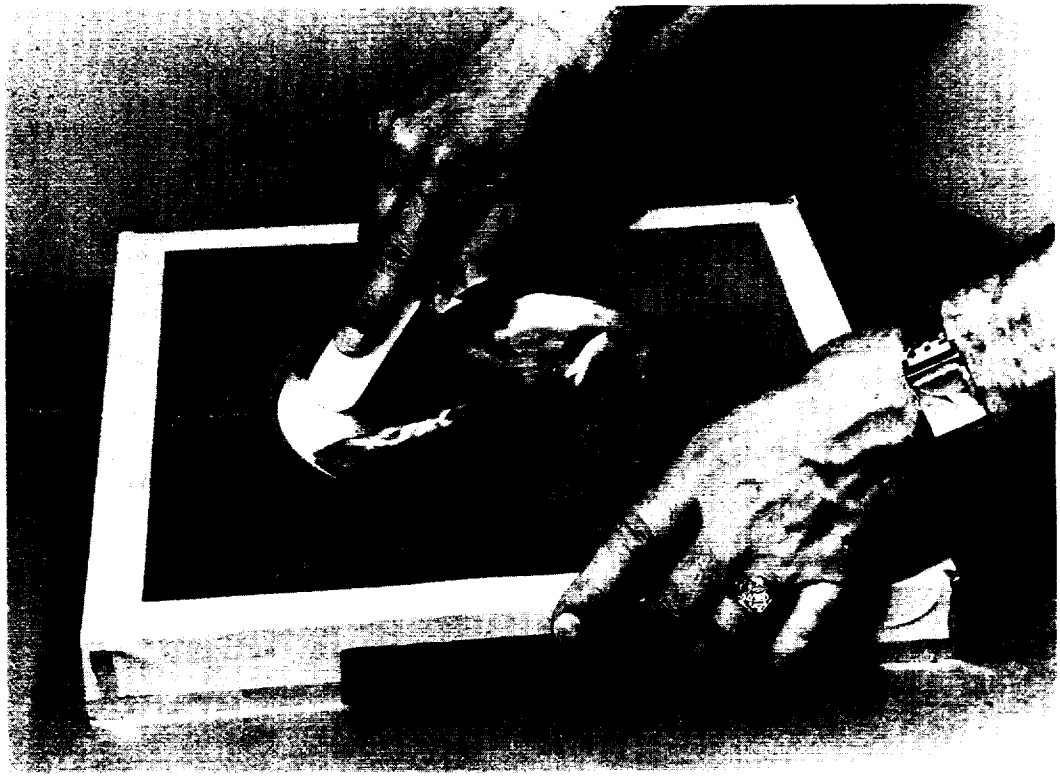


Figure 20. Stepped Nickel Liner



a. Forming with Teflon Tool

b. Removal of Center Blank

Figure 21 End-Flange Liner Fabrication

TABLE VII

FABRICATION VARIABLES AND TESTING RESUME

Vessel	Max. pressure	Type test	Failure	Longitudinal reinforcement	Scrim cloth in longitudinal joint	Longitudinal doubler	Temp., °F	Adhesive	Scrim	Composite stress (ksi)
D1	569	Burst	Liner seam	No	No	Yes	-423	100% L-100	No. 112 glass	142.0
D2	815	Burst	Liner seam	No	Yes	Yes	-423	100% L-100	Glass	192.0
TA-1	711	Burst	Longitudinal failure	No	Yes	Yes	-423	70:30(L-100:Epi-Rez 5101)	Glass	179.0
TA-2	403	Cyclic	Longitudinal glass displaced; vessel failed	No	Yes	Yes	-423	70:30	Glass	98.3
TA-3	550 (580)	Cyclic	Cycled 100 times; no failure; leak on burst	Yes	Yes	Yes	-423	70::30	Glass	135.0 (142.5)
TA-4	550	Cyclic	Cycled 20 times decreasing from 550 psi to 395 psi; liner leaked	Yes	Yes	Yes	-423	70:30	Glass	135.5
TA-5	514	Burst	Hoop-longitudinal; glass failure	Yes	Yes	No	+75	70:30	Glass	126.5
TA-6	500	Cyclic	Cycled 23 times; liner leaked	Yes	No	No	+75	70:30	Glass	123.8
TA-7	500	Cyclic	Cycled 41 times; liner leaked	Yes	Yes	No	+75	70:30	Glass	122.0
TA-8	630	Burst	Longitudinal lamination in transition	No	Yes	No	+75	70:30	Glass	155.0
TA-9	678	Burst	Longitudinal failure in transition	No	Yes	No	+75	70;30	Glass	167.5
TA-10	774	Burst	Longitudinal failure in transition	No	No	No	+75	70:30	Glass	192.3
TA-11	500	Cyclic	Cycled 40 times; liner leaked	No	No	No	+75	70:30	Glass	122.0
TM-1	574	Burst	Liner failure in fiberglass transition and nickel transition	No	--	--	+75	70:30	A2951 Nylon (A)	141.0
TM-2	--	--	Liner and vessel destroyed during mandrel removal	--	--	--	--	--	--	--
TA-12	500 545	Cyclic	Cycled 5 times at +75°F; failed first cycle -423°F	No	No	No	+75 -423	G207	None	120.0 130.5
TA-13	500	Cyclic	Cycled 2 times at +75°F; failed at 490 psi 3rd cycle	No	No	No	+75	G207	None	119.3
TA-14	500 550	Cyclic	Cycled 5 times at +75°F; 7 small leaks sealed; cycled 10 times at -423°F	No	No	No	+75 -432	80:20(L-100: Epi-Rez 5101)	34168-2 Nylon (B)	119.3 131.1
TA-15	500 550	Cyclic	Cycled 5 times at +75°F; one nondetectable leak; cycled 10 times at -423°F[a]	No	No	No	+75 -423	80:20	Nylon (B)	120.8 133.0
TA-16	500 550	Cyclic	Cycled 5 times at +75°F; no leakage; cycled 10 times at -423°F; slight leakage caused low pressure cycles[a]	No	No	No	+75 -423	80:20	Nylon (B)	118.9 130.9
TA-17	500 550	Cyclic	Cycled 5 times at +75°F; no leakage; cycled 10 times at -423°F to 392 psi, 10 times at -423°F to 550 psi[a]	No	No	No	+75 -423	70:30	Nylon (B)	118.9 130.9
TA-18	500 550	Cyclic	Cycled 5 times at +75°F; no leakage; cycled 10 times at -423°F; very slight seam leakage[a]	No	No	No	+75 -423	70:30	Nylon (B)	119.8 131.8
TA-19	500 550	Cyclic	Cycled 5 times at +75°F; leakage occurred on the 5th cycle[a]; sealed; cycled 10 times at -423°F; leakage caused 3 low pressure cycles.	No	No	No	+75 -423	G207	None	120.8 133.0
TA-20	500 550	Cyclic	Cycled 5 times at +75°F; no leakage; cycled 10 times at -423°F; leakage caused 3 low pressure cycles	No	No	No	+75 -423	70:30	Nylon (B)	120.6 133.0
TA-21	500 550	Cyclic	Cycled 5 times at +75°F; one liner leaked; sealed cycled 10 times at -423°F leakage caused 4 low-pressure cycles	No	No	No	+75 -423	G207	None	118.5 130.4

[a]Primary liner O.K. Seam leaked.

Development vessel D1: This vessel was fabricated with an aluminum foil liner. The aluminum was bonded with 100% Adiprene L-100/MOCA with No. 1122 E glass A1100 finish cloth used as the scrim (vessel fabrication was completed before final selection of the most-promising adhesive from Task I). No scrim was used in either the circumferential or longitudinal joints. Scrim was used in the circumferential and longitudinal doublers. Annealed 6-mil aluminum foil was formed as end-flange liners. Aluminum (0.032-in.-thick 7075-T6) stiffening rings were used in the flanges (fig. 22a). The flanges were fabricated by a wet lay-up technique.

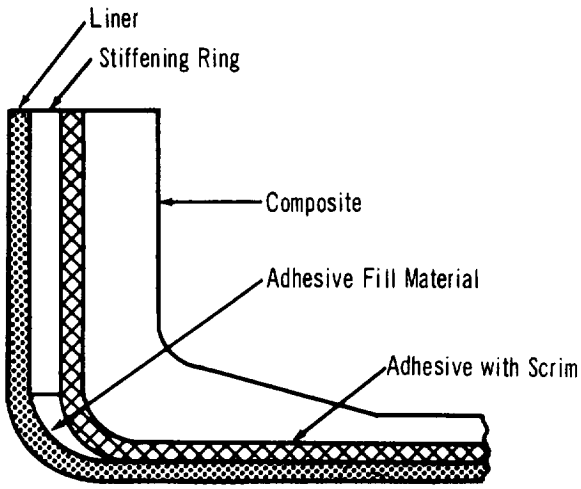
Development vessel D1 was pressure tested at a temperature of -423°F. The vessel attained a maximum hoop strain of 2.1% at an internal pressure of 569 psi, at which time the vessel leaked excessively. Stress-strain curves for the vessel are given in figure 23. The desired objective of hoop to longitudinal strain ratio of 1/1 in the test section was achieved.

The internal pressure was applied to the vessel at a rate to cause a strain of approximately 1%/min. to 130 psi. At this point, all vacuum was lost in the chamber. The internal pressure was increased to a maximum of 569 psi at a rate which caused the vessel to strain approximately 4%/min. The vessel did not burst.

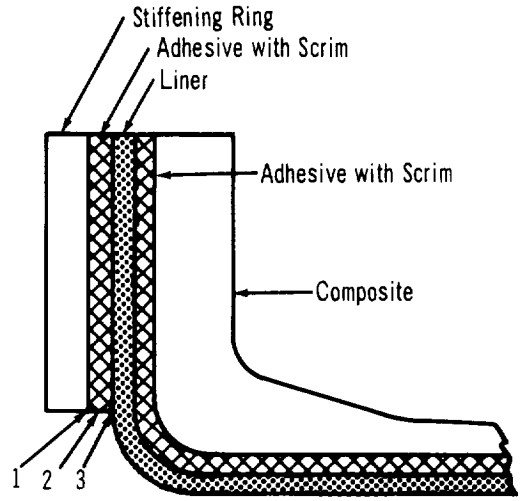
One set of strain measurements was made with electrical resistance strain gages (modified Karma SK-15-125-TM-350*) bonded to the vessel interior with BR-600 adhesive*. Another set of strain measurements was made by connecting deflection wires to the specimen and strain beams. No test data were obtained from the strain gages because they debonded from the liner at a low internal pressure. Previously to the test of vessel D1, the positive feasibility of internal strain gages was demonstrated in a test system checkout. During the system checkout, strain gages applied to a nickel-lined cylinder responded to pressure changes and stayed bonded to 400-psi internal pressure at -423°F. (This was the maximum pressure attempted.)

A post-test ambient leak check of the vessel at 20 psi showed leakage in the longitudinal doubler area. A small delamination (1/4 x 1/2 in.) was found in the area which exhibited the leakage observed in the ambient leak check. The aluminum liner in the cylindrical section, except for the area of the internal longitudinal doubler, appeared to be completely bonded. There were no apparent voids or delaminations. The bond line between the doubler and the liner contained voids. In some cases, liquid was probably forced behind the doubler into the void areas and upon release of pressure, the pressure differential caused a balloon effect. Doublers had been used in both the longitudinal and circumferential joints.

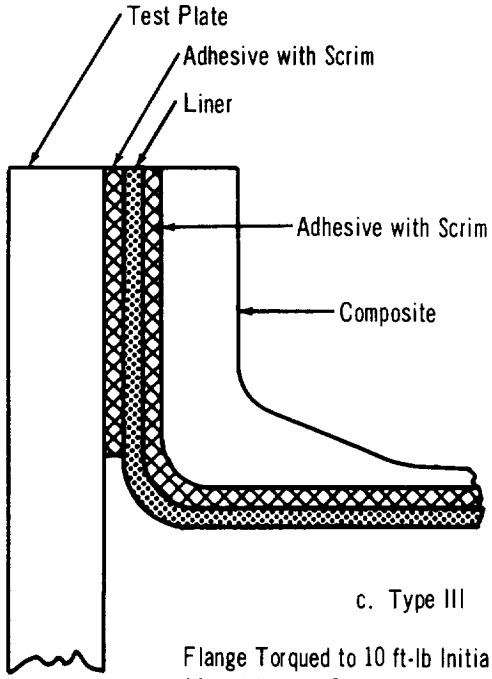
* Manufactured by Micromeasurements, Inc.



a. Type I



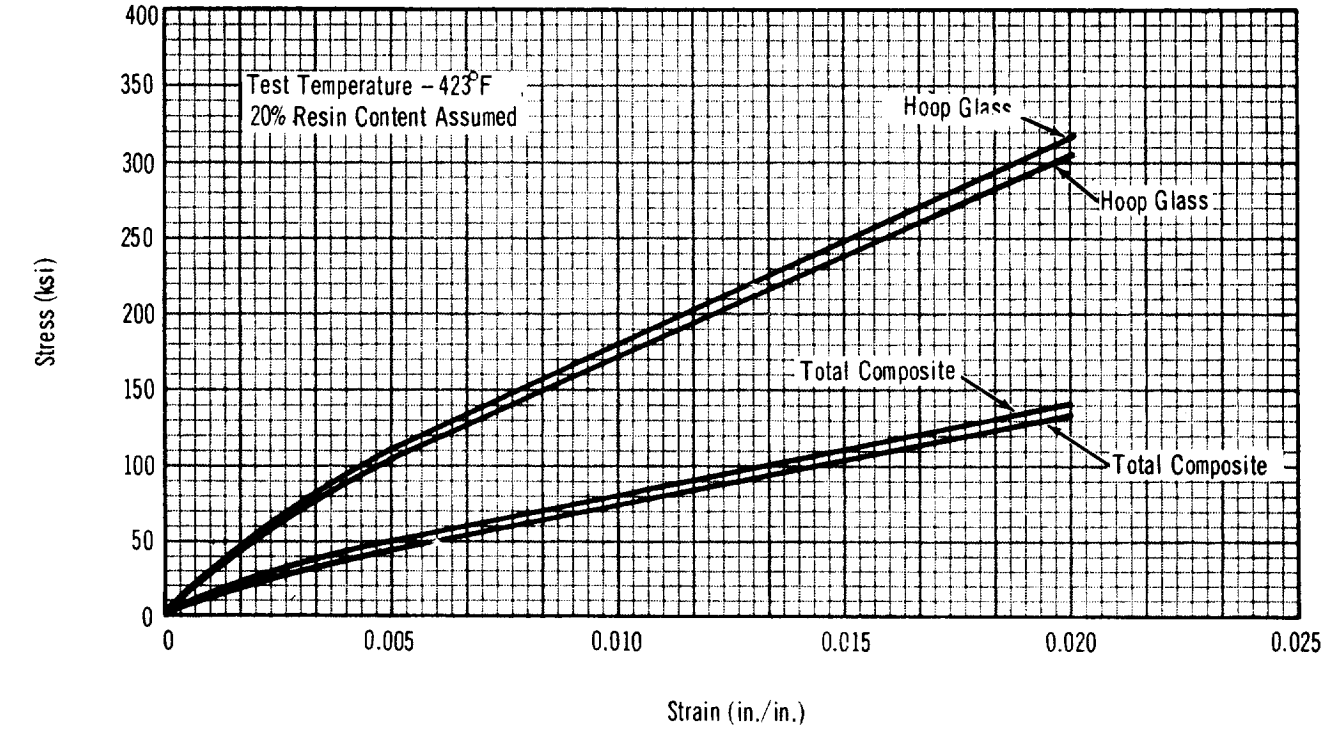
b. Type II



c. Type III

Flange Torqued to 10 ft-lb Initially. Torqued to 100 ft-lb After Adhesive Cures

Figure 22. Flange End-Seal Methods



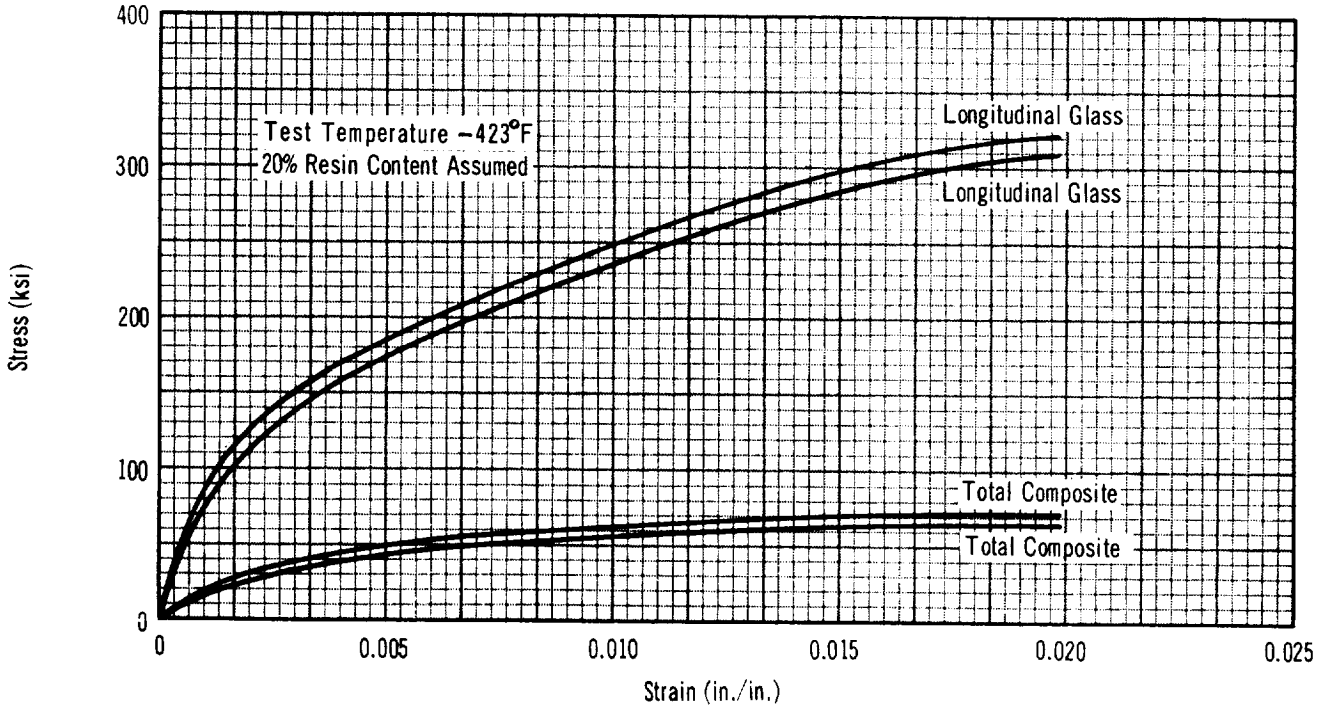


Figure 23. Stress-Strain Diagram, – 7 1/2-in.-Diam Vessel D1

Details of the test system, procedure, and NAFLEX end seals are described in pertinent sections of the Task III discussion.

Development vessel D2: This vessel was fabricated similarly to vessel D1. Improvement was expected in sealing by the use of scrim cloth in all joints and doubler areas and a reduction in the width of the doublers.

The fiberglass lay-up was identical to that of vessel D1, except that the flange was fabricated with preimpregnated glass cloth. The vessel was tested at -423°F. Maximum pressure reached 815 psia. Excessive leakage through the liner doubler-joint and/or end seal prevented continued pressurization. A maximum hoop strain of 3% was attained. The stress-strain curve is shown in figure 24.

The two biaxial strain gages did not stay bonded to the aluminum liner. One fell off during chilldown and the second fell off very early (at less than 50-psi internal pressure) during the pressurization. The aluminum liner had been sand-blasted in the installation area before strain gage installation.

Post-test inspection revealed that the liner in all but the longitudinal doubler area looked excellent. There were no cracks, wrinkles, or buckles. The circumferential doublers appeared satisfactory. There were some voids that appeared as indentations but no cracks, bubbles or debonding.

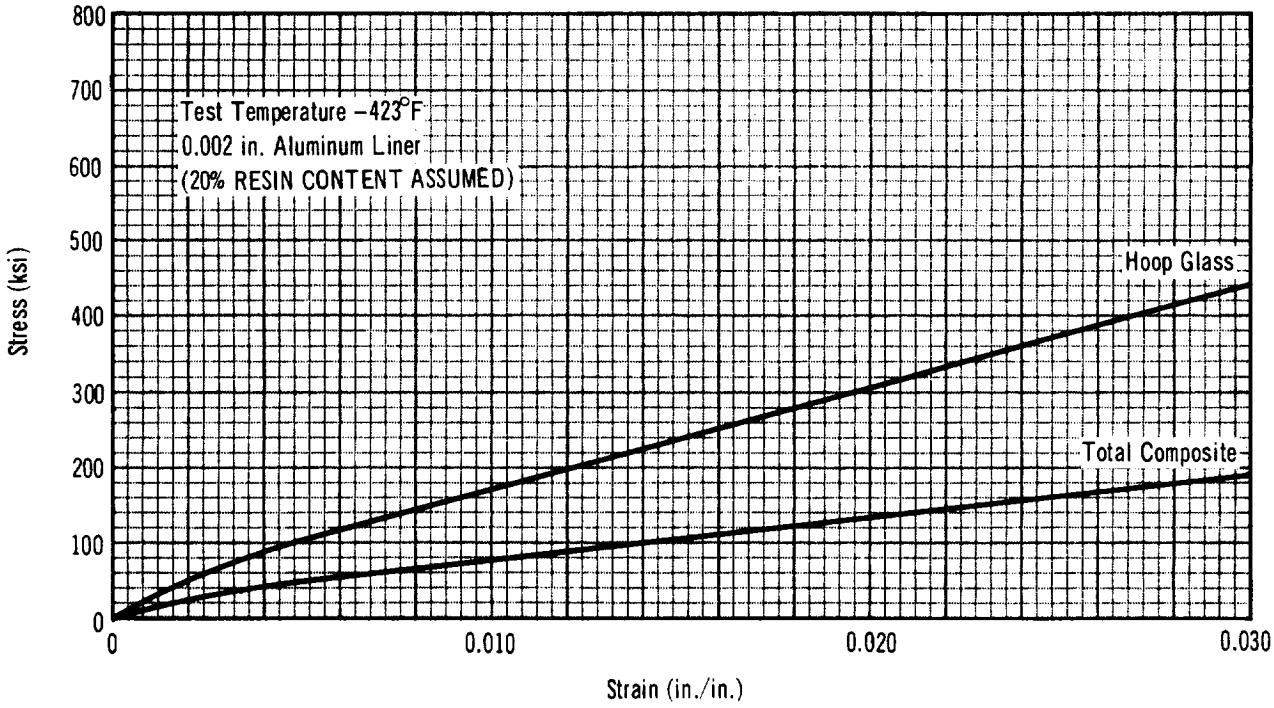


Figure 24. Hoop Stress-Strain Diagram – Vessel D2

In the seal area at the inner edge of the backup ring, cracking of the foil occurred. At one end there were two cracks, one was 3/4-in. long and the other was 1/4-in. long. At the other end there was one crack 3/4-in. long. The longitudinal doubler contained voids that appeared as delaminations and bubbles in the test section, particularly along the edges (fig. 25). Cracking of the doubler occurred in some of the delaminated buckled areas.

Test Vessel Fabrication. - Aluminum-lined vessels were fabricated first. Nickel-lined vessel fabrication was concurrent with the fabrication of the 9th and 10th aluminum-lined vessels.

The adhesive that was used in vessel fabrication was the Task I selected adhesive system, L-100:5101 (70:30)/MOCA with glass scrim, unless otherwise noted.

Vessel TA-1: The first vessel, TA-1 (Test vessel, Aluminum liner, No. 1) was fabricated with the liner at the flat of the flange unbonded to the aluminum stiffening ring, as in development vessel D2, to ensure a smooth, flat, void-free area for the NAFLEX seal (Task III). The internal doubler, installed after the resin system was cured, was approximately 1-in. wide and it was scalloped (see fig. 26) to improve strain intrusion to the doubler. Other construction details were the same as development vessel D2.

During the fabrication of the vessel, the initial overwrapping of the build-up resulted in a displacement of the longitudinal tape at one end, approximately 1 in. in the build-up. The displacement was approximately 3/8 to 1/2 in., in a distance of 2 in.

Vessel TA-2: Vessel TA-2 incorporated a new type of end seal (see fig. 22b). The stiffening ring which had been previously used behind the liner was brought in front of the liner. Other construction details were the same as vessel TA-1.

During the initial overwrapping of the end buildup, the tension of the hoop tape was reduced and the excess "buttering-up" resin was eliminated to avoid displacing the longitudinal tape. Some displacement was evidenced (approximately 1/8-in. in 1-1/2 to 2 in. of length). The displacement was apparently minor, so fabrication of the vessel was completed.

Vessel TA-3: This vessel was fabricated with the flange construction shown in figure 22c. This differed from that of vessel TA-2 because of the unavailability of the NAFLEX test seals (damaged during a previous test).

To avoid the longitudinal glass displacement that had occurred with vessels TA-1 and TA-2, the vessel was fabricated to the initial overwrapping point and then allowed to partially polymerize. Also, to aid in transferring the load into the build-up, an additional 3-in. length of longitudinal glass tape was used in the build-up areas over the longitudinal sheet.

Figure 25. Vessel D2. Post-Test, General View

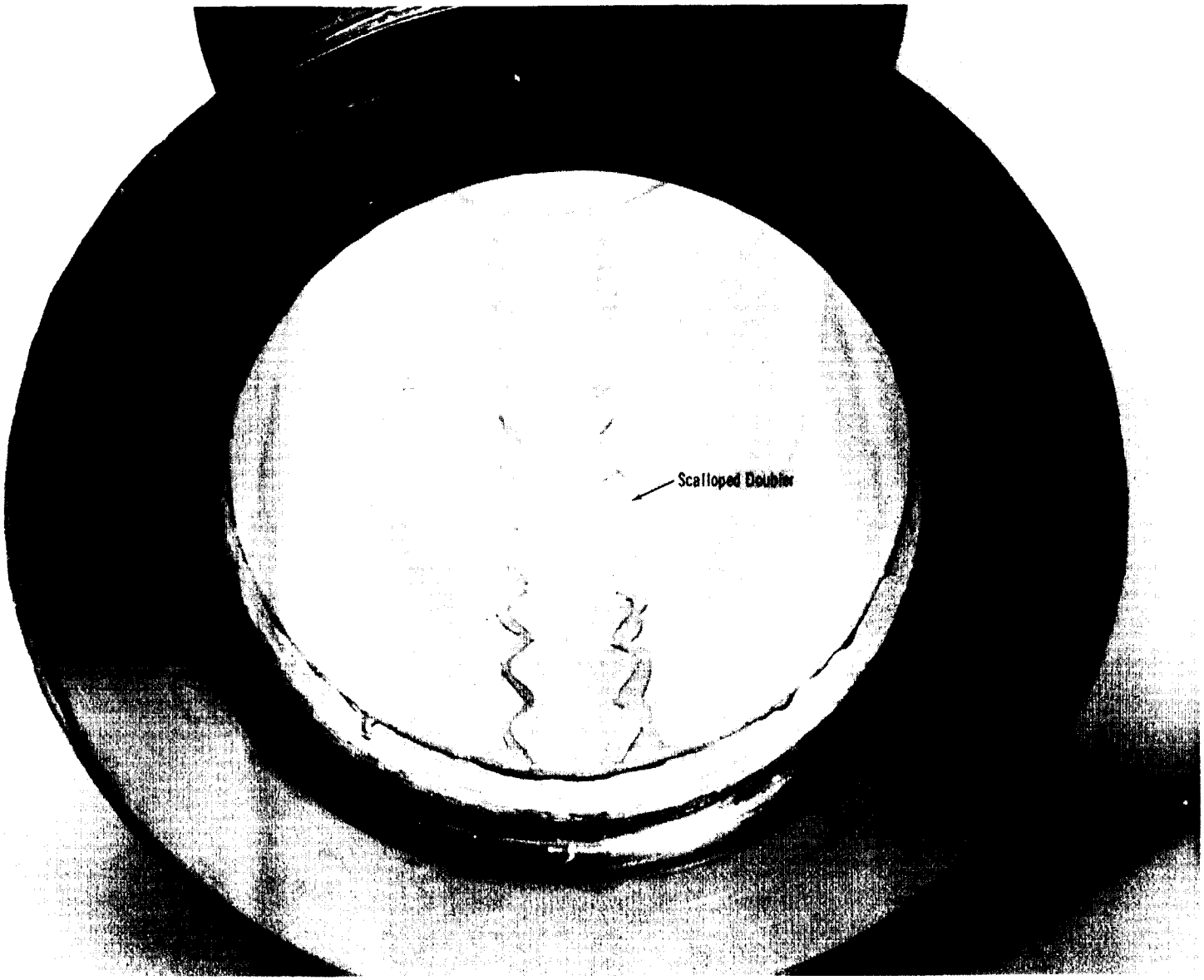


Figure 26. Vessel TA-2, Pretest, Vessel Interior

Other construction details were the same as vessel TA-2.

Vessel TA-4: Flange construction of this vessel was the same as that of vessel TA-3. Vessel TA-3 was satisfactorily sealed during a -423°F cyclic test and this, together with its ease of fabrication, recommended it for vessel TA-4 and future cryogenic test vessels.

A 4-in. length of glass tape was used in the build-up area under the longitudinal tape to help in transferring the longitudinal load into the build-up. This tape was used in addition to the 5-in. length used over the longitudinals. (A 3-in. long tape was used over the longitudinals in vessel TA-3. The additional 2-in. of tape used in this vessel were considered worthwhile to increase the transference of load, even though fabrication difficulties were increased by use of the greater length.)

Other construction details were the same as vessel TA-2.

Vessel TA-5: This vessel was fabricated similarly to vessel TA-4. The use of the longitudinal doubler was discontinued because it apparently contributed nothing to vessel performance.

Vessel TA-6: Vessel construction was the same as TA-5, except that no scrim cloth was used in the longitudinal joint.

Vessel TA-7: This vessel was fabricated similarly to vessel TA-6.

Vessel TA-8: Vessel construction was similar to TA-5, except that no longitudinal strip reinforcement was used. The use of the circumferential doubler was discontinued.

Because of an early longitudinal displacement problem, which was manifested severely on vessel TA-2, longitudinal reinforcement was placed over the longitudinals leading into the build-up area; this was done to aid the load transfer into the buildup. With this, a reduction in tape tension during the start of hoop winding, starting closer to the flange, and higher B-staging during hoop wrapping were made with vessels TA-4 through TA-7. It appeared from a physical examination of the tested specimens and a vessel review, that the inclusion of the longitudinal reinforcement may have helped transfer the load in the build-up area, as intended, but the discontinuity it caused at the start in the test section apparently created a far more serious problem, i.e., high concentrated strain. The use of the short longitudinals was discontinued.

The cure of 7-hr. at 250°F was interrupted after 3-1/2 hr. when the curing oven became inoperative. The vessel was then reheated to 250°F and the cure continued for the additional 3-1/2 hr.

Vessel TA-9: Vessel construction was similar to TA-8.

Vessel TA-10: Vessel construction was similar to TA-8, except that no scrim cloth was used in the longitudinal and circumferential seams. Also, the single layer of longitudinal tape was placed directly on the first hoop wrap layer. This placed the longitudinal layer in single shear rather than double shear (see fig. 27).

Vessel TA-11: Vessel construction was similar to TA-10, except that a nylon scrim cloth (A2951/c) was used in place of the previously used No. 122 glass cloth. The nylon was used in this vessel to evaluate its ambient temperature cyclic resistance compared to the basic system.

Fabrication of nickel lined vessels: Three liners were supplied under the Lockheed development program; one was a preliminary test item and the other two, the subcontracted items.

Quality control coupons cut from the Q. C. drum of the three mandrels had average tensile elongations of 9-1/2, 16 and 16%, respectively, of approximately 0.004-in. material (desired value was minimum of 12%). The preliminary test liner was stripped from the mandrel (liner had sustained a tear during fabrication by Lockheed) and a thickness profile (fig. 28) was made of the scrapped liner. Tensile coupons were cut from the 12-in. test section; the average elongation of eight specimens was 8%, which corresponded with the Q. C. test coupons.

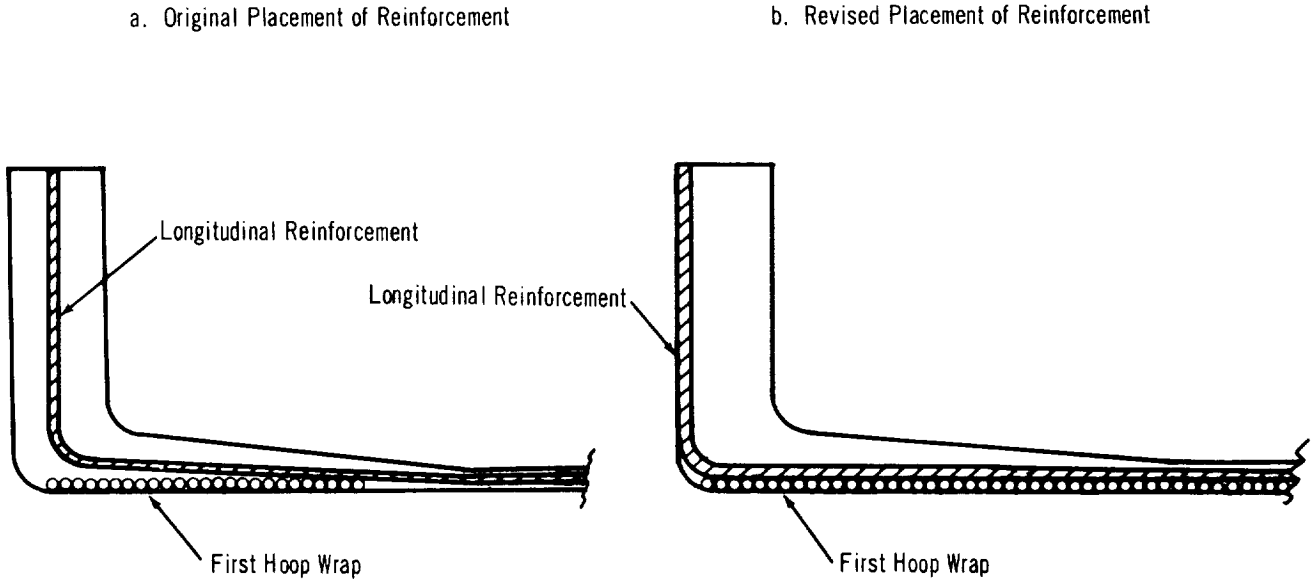


Figure 27. Longitudinal Reinforcement Positioning

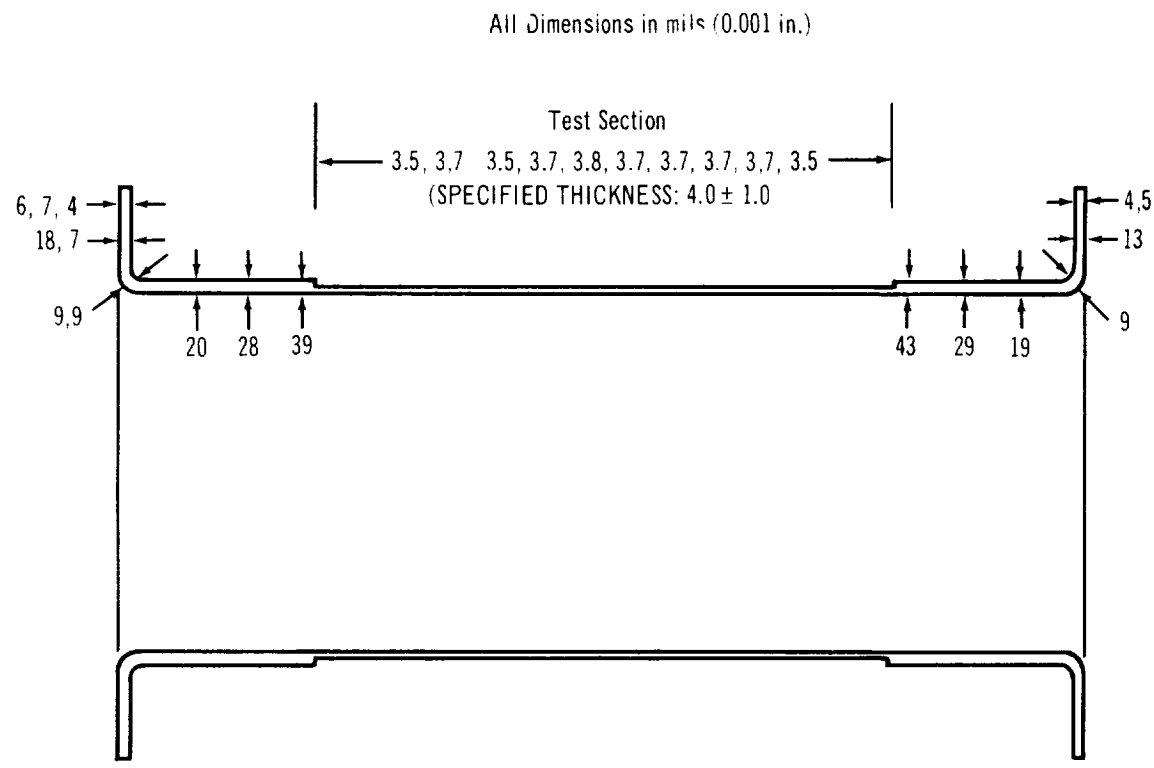


Figure 28. Thickness Profile, Preliminary Electro-Deposited Test Liner

Vessel TN-1: This vessel was fabricated with the third liner deposited by Lockheed. Nodules at the transition section were manually filed to avoid cutting the fiber glass. The vessel was fabricated similarly to aluminum-lined vessel TA-8.

Removal of the mandrel following vessel cure was extremely difficult. The procedure that proved successful was to wrap the vessel with heating tape, and chill the mandrel interior with dry ice. During mandrel removal, the liner was torn for a distance of approximately 1-in. long by 1/2-in. wide, at a point where the deposition had bonded to the stainless steel mandrel (fig. 29). The liner tear was repaired with aluminum foil overlay.

Vessel TN-2: This vessel was fabricated similarly to vessel TN-1. However, following vessel cure, the mandrel could not be removed from the vessel. The nickel had been torn and peeled back from the vessel during removal of the end plate. Subsequent removal of the liner from the mandrel (following destruction of the fiberglass shell) proved to be extremely difficult, even with the use of a wooden chisel to effect peeling action.

Lockheed personnel indicated that future vessel removal could be guaranteed. Obviously, the passivation of the mandrel surface had not been complete with the development liners. Future passivation processing could include a chromic acid immersion, nitric acid immersion, and hot water rinse.

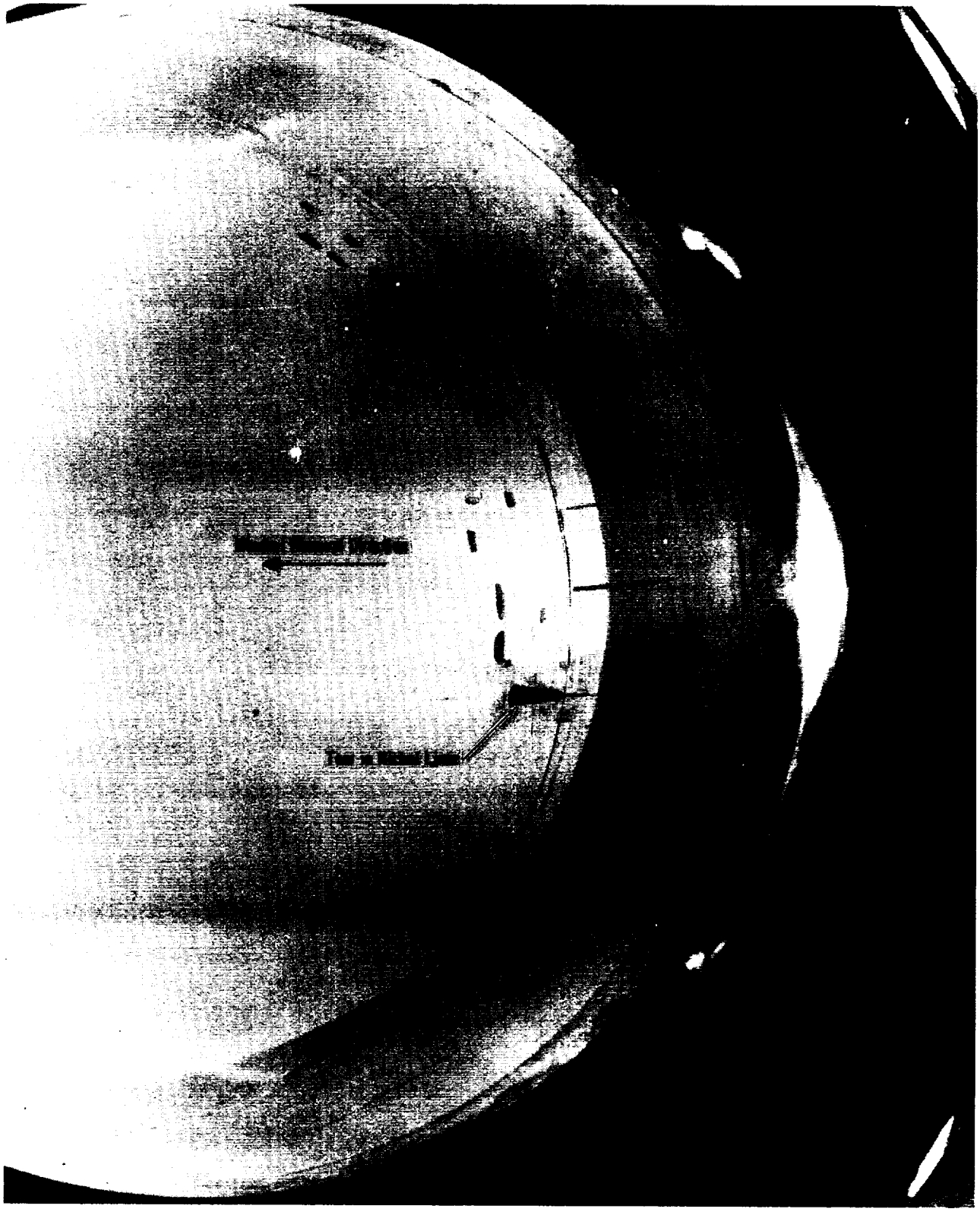


Figure 29. Vessel TN-1, Tear in Nickel Liner

These items, together with a fast strike of nickel in the planting tank, before full production "hook-up" could provide the necessary steps for non-adherance of the electroplate to the stainless steel mandrels. However, at that time in the program, the emphasis was given to an evaluation of additional adhesive systems (L-100:5101 (70:30)/MOCA and nylon scrim, L-100:5101 (80:20)/MOCA and nylon scrim, and G-207 (no scrim) with aluminum liners rather than continuing with the L-100:5101 (70:30)/MOCA and glass scrim system with nickel liners. The nickel liner work was discontinued.

Continuation of aluminum-lined vessel fabrication:

Vessel TA-12: This vessel was fabricated with the Goodyear polyester adhesive, G-207, without a scrim cloth. Vessel construction was similar to TA-9 in that the longitudinal glass was placed through the flange buildup, rather than directly over the first hoop layer as in TA-10. The earlier, less desirable, vessel construction was used because of a change in fabrication personnel and an incompletely revised fabrication order.

Vessel TA-13: This vessel was also fabricated with G-207 without scrim. Vessel construction was similar to vessel TA-10.

Vessel TA-14: This vessel was fabricated with Adiprene L-100:Epi-Rez 5101 (80:20)/MOCA with nylon scrim adhesive system. Vessel construction was similar to vessel TA-13. Slight longitudinal glass slippage was noted during the wrapping of the second hoop layer. Consequently, future vessels were fabricated with the area of second hoop wrap initiation highly advanced in resin cure.

Vessel TA-15: This vessel was fabricated with the 80:20 mixture adhesive. Construction was similar to vessel TA-14, except that the area of second hoop wrap initiation was highly advanced to prevent slippage of the underlying longitudinal layer. Tension for this and subsequent vessels was reduced from 1/2 to 1/4 lb per end of glass.

Vessel TA-16: This vessel was fabricated with the 80:20 mixture adhesive. Construction was similar to vessel TA-15.

Vessel TA-17: This vessel was fabricated with the Adiprene L-100:Epi-Rez 5101 (70:30)/MOCA with nylon scrim adhesive system. Vessel construction was similar to vessel TA-15.

Vessel TA-18: This vessel was fabricated with the 70:30 mixture adhesive. Construction was similar to vessel TA-15.

Vessel TA-19: This vessel was fabricated with G-207 without scrim. Vessel construction was similar to vessel TA-15.

Vessel TA-20: This vessel was fabricated with the Adiprene L-200:Epi-Rez 5101 (70:30)/MOCA with nylon scrim adhesive system. Vessel construction was similar to vessel TA-15.

Vessel TA-21: This vessel was fabricated with the G-207 adhesive, without scrim. Vessel construction was similar to vessel TA-15.

Task III - Pressure Vessel-Liner Evaluation Tests

The objective of Task III was to determine the cycling capability of nickel and aluminum liners in filament-wound vessels at ambient and liquid hydrogen temperatures. The first phase of work consisted of the following. One vessel was to be made with each liner and pressurized to failure and three others were to be cycled 100 times to 2% strain at ambient temperature. One vessel was to be made with each liner and six others were to be cycled 100 times to 2% strain at -423°F. Thirteen tests were completed. Liquid nitrogen testing, a part of the original program, was deleted because greater emphasis of effort and expenditure was needed on the nickel work in Task II. Subsequent developments led to the cancellation of the production nickel effort together with a redefinition of the remainder of the program.

The second phase of work in Task III consisted of testing 10 vessels. The procedure was as follows. Each vessel was to be pressure-cycled five times from 0 to 500 psi at +75°F or until failure, whichever occurred first. The specimen was then to be removed from the chamber and an inspection made of the liner. The vessel was then to be reinstalled in the chamber and pressure cycled 10 times from 0 to 550 psi at -423°F or until failure, whichever occurred first. The specimen was then to be removed from the chamber and an inspection made of the liner.

Facility and Instrumentation. - Each vessel test was conducted in the Propulsion Laboratory Cryogenic Facility. The ambient temperature test system and cryogenic temperature test system are shown in figures 30 and 31, respectively.

All of the first-phase test vessels, both burst test and cyclic test (vessels D1, D2, TA-1 through TA-11, and TN-1), were instrumented with four longitudinal deflection gages and two circumferential growth deflection gages to permit the acquisition of longitudinal and circumferential strain data. Biaxial strain gages were also installed on the interior of vessels D1, D2, and TA-1, but further use of strain gages was discontinued when no useful data were obtained.

The second-phase test vessels were not instrumented with either deflection gage or strain gage instrumentation.

Seals. - All ambient temperature vessels were sealed with teflon-impregnated gaskets or rubber gaskets.

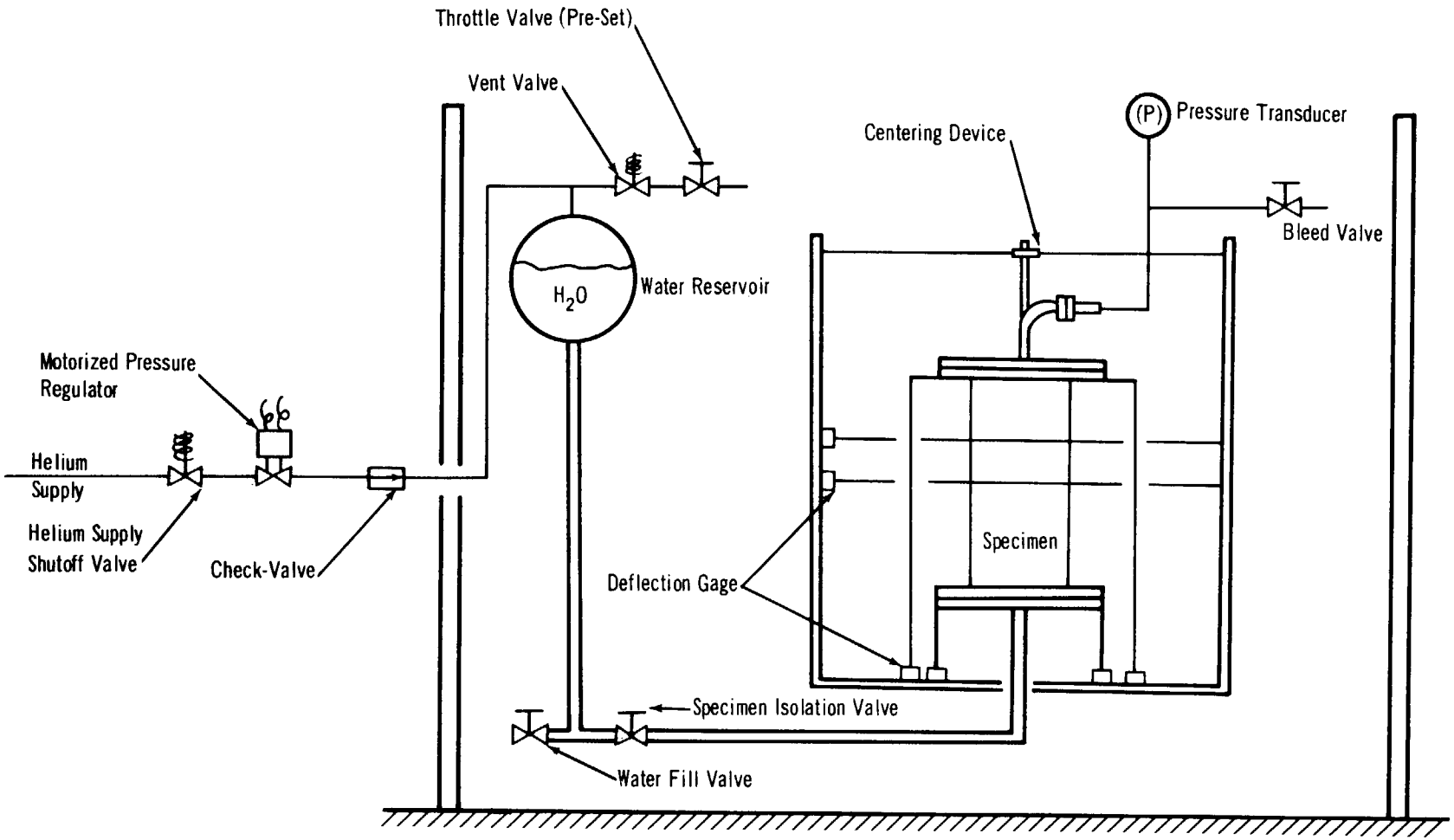


Figure 30. Ambient Temperature Test System Schematic

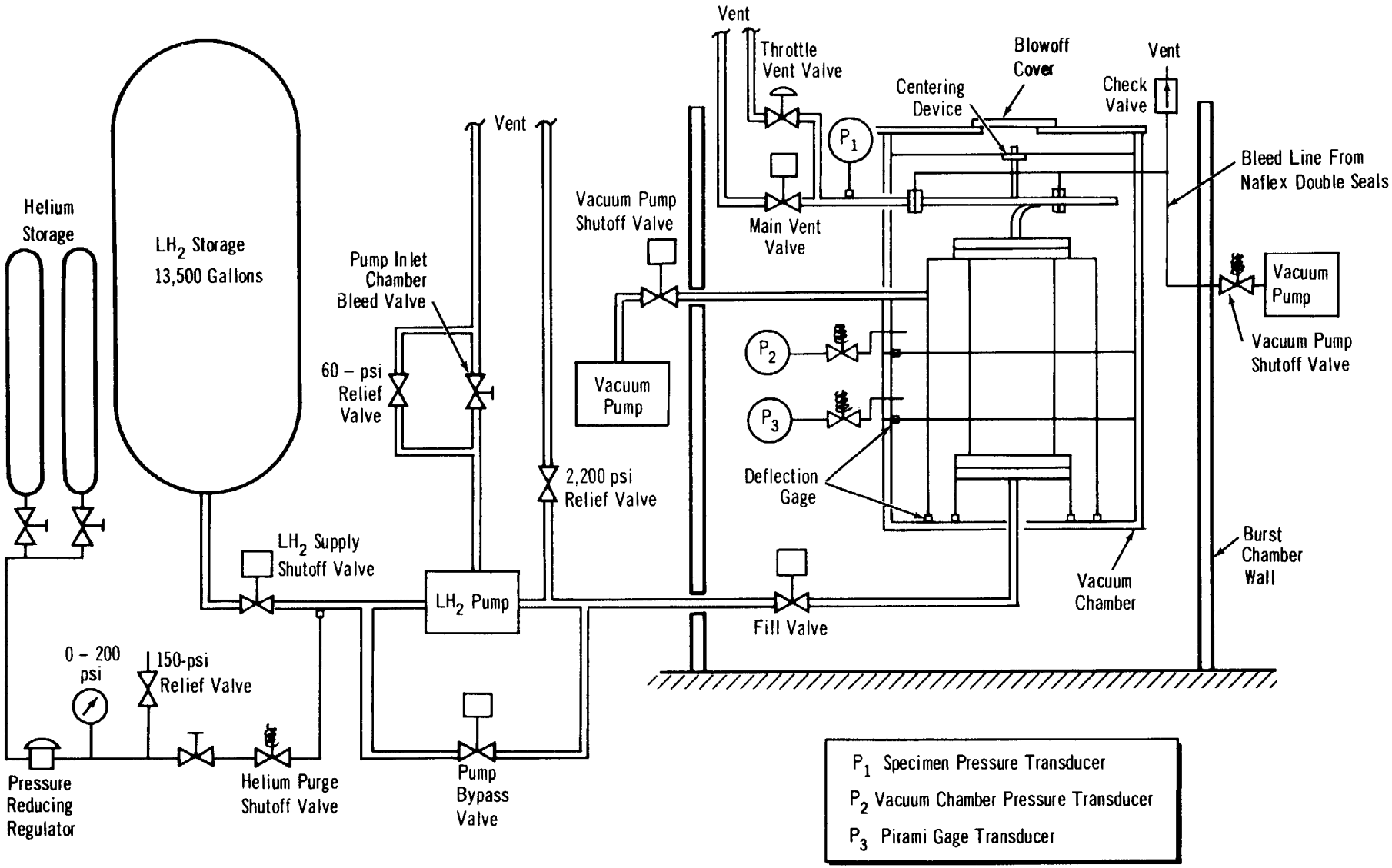


Figure 31. Liquid Hydrogen Test System Schematic

Several of the initial first-phase test vessels were sealed with NAFLEX seals* (fig. 32). The use of the evacuated seal established that the leakage into the vacuum chamber was through the liner exclusively. The seal performed well in the two vessels so instrumented. However, the teflon coating on the sealing surfaces of each of the two sets of seals was damaged during the burst tests. The time which would have been involved in refurbishing the seals would have caused gross schedule slippage, so a new method of sealing was sought. The solution, which turned out to be successful from all standpoints, was the sealing method shown in figure 22c. That is, the test plates were simply bonded to the liner-vessel flange with 100% polyurethane adhesive (L-100/MOCA) and No. 112 glass cloth.

Procedure. - The detailed test procedure is given in Appendix D. Briefly, each vessel was leak tested before and after each test. Burst tests were made by pressurizing the vessels until failure. Cyclic tests were made by pressurizing the vessel to the pressure required to strain the vessel to 2.0% as identified from the burst tests. All vessels were pressurized at a rate to cause a strain rate of less than 1%/min.

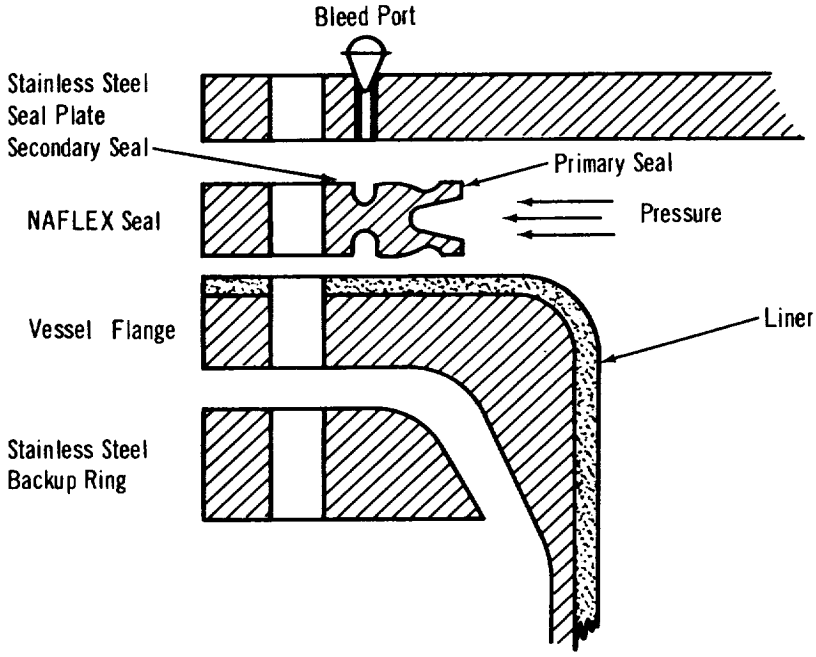


Figure 32. NAFLEX Seal

*Manufactured by Navan Products, Inc.

Phase I Testing. - Each vessel was tested at a single temperature. A compilation of test results is given in table VII.

Vessel TA-1: This vessel was tested at -423°F. It failed at a pressure of 711 psi.

Deterioration of vacuum chamber pressure and bleed line pressure across the NAFLEX seal increased at essentially the same time which indicated that vacuum chamber pressure rise was caused by a leaking end seal and not a liner leak. The strain gages became inoperative early in the test.

Post-test inspection of the vessel revealed that failure occurred in the longitudinal glass in the build-up area. The failure did not occur in the build-up end which had displaced longitudinals. The vessel is shown at post-test in figure 33. The stress strain curve is shown in figure 34.

The primary liner remained excellent; there were no voids, cracks, wrinkles or buckles.

Vessel TA-2: This vessel was tested at -423°F. End-seal configuration was of the type shown in figure 22b.

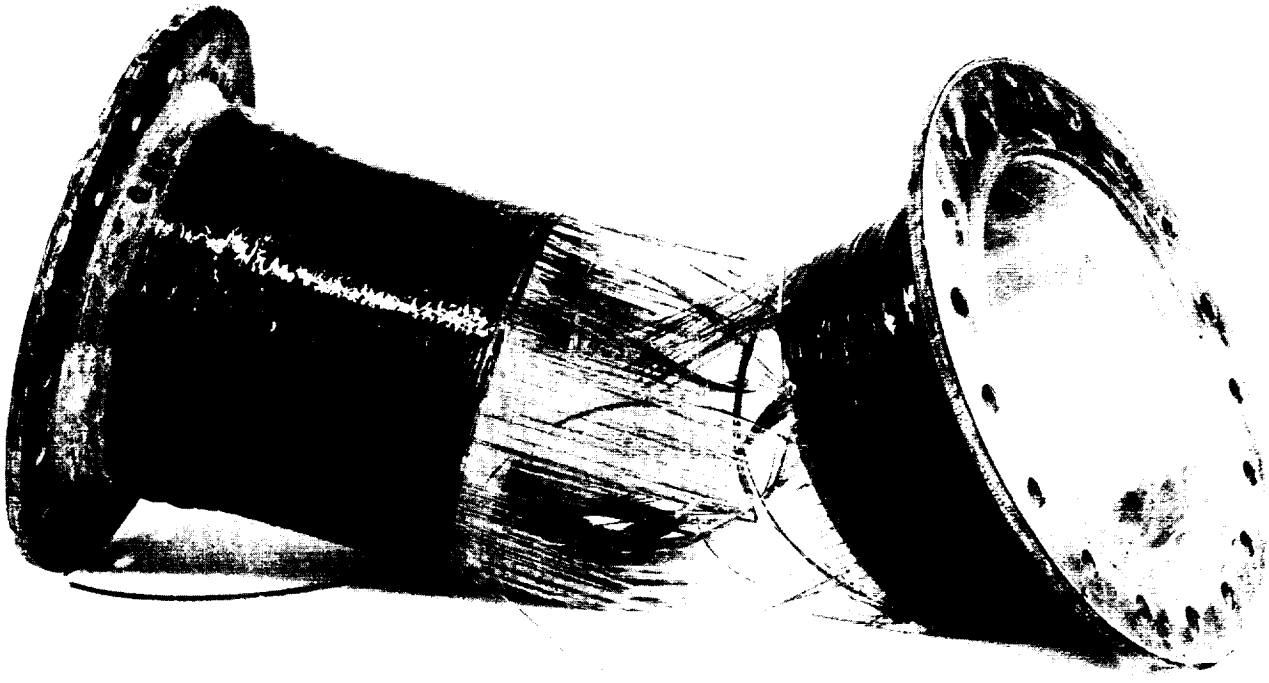


Figure 33. Vessel TA-1–Post-Test, General View

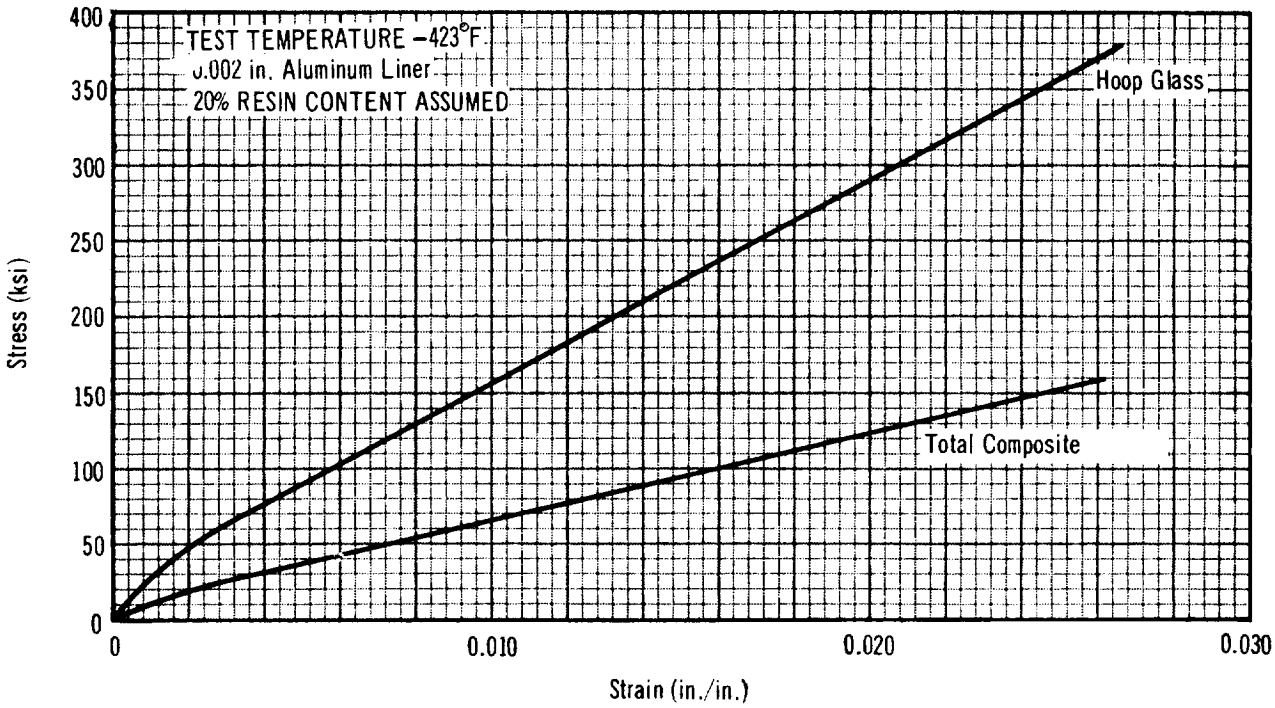


Figure 34. Vessel TA-1 Hoop Stress-Strain Diagram

The vessel remained relatively well-sealed throughout the test. No apparent leakage occurred across the NAFLEX seal.

The vessel failed on the initial intended cycle to 550 psi at 403 psi.

Post-test inspection of the vessel revealed that failure occurred in the longitudinal glass because of a displacement, caused by the hoop overwinding in the build-up area. The displacement was much more severe than noted during the fabrication. The vessel is shown post-test in figures 35 and 36. During the fabrication of the vessels from TA-3 onward special attention was paid to eliminating the displacement problem.

The primary liner remained satisfactorily bonded throughout the vessel.

Vessel TA-3: This vessel was cyclic tested at -423°F. End-seal configuration was of the type shown in figure 22b. The vessel was cycled to 2% strain at 550 psi for 101 cycles. This was followed by two attempted burst cycles. The maximum pressure attained on the burst attempts was 580 psi. Because of excessive leakage, the vessel could not be burst. The post-test leak check revealed three areas of leakage near the liner longitudinal seam. No leakage was detected at or near the bonded-on flange plate.

Figure 35. Vessel TA-2–Post-Test Overall View

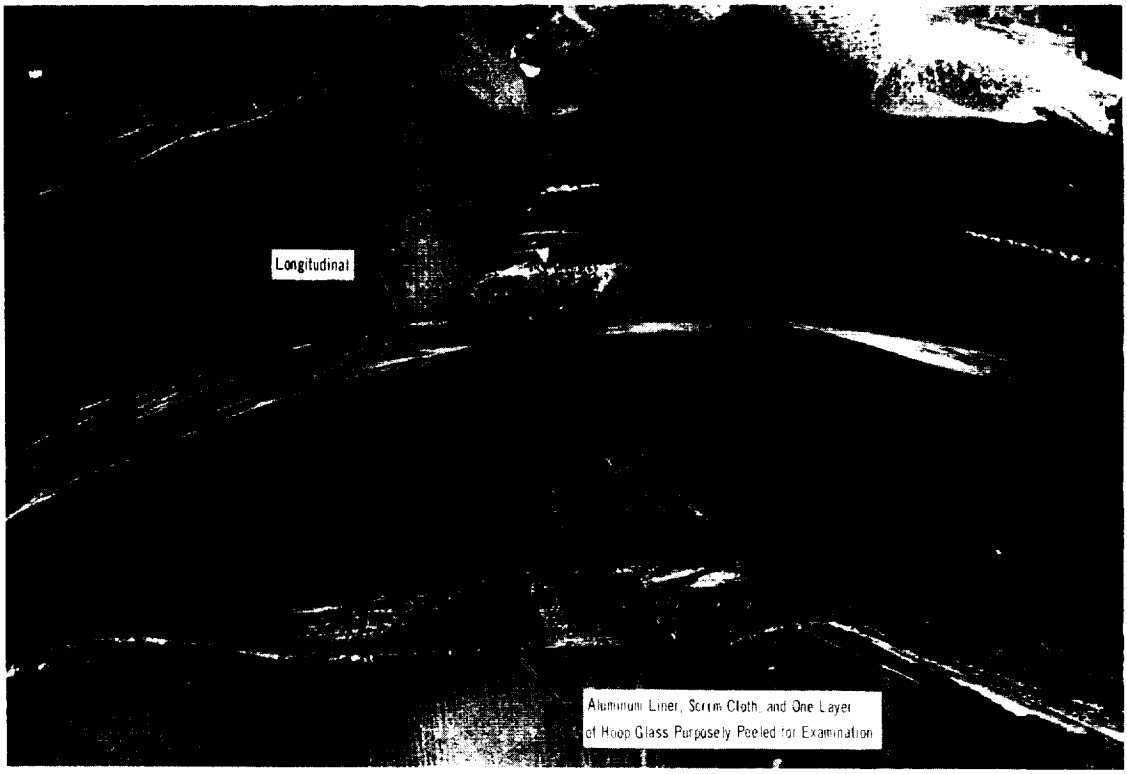


Figure 36 Vessel TA-2 – Post-Test Closeup View of Failure End

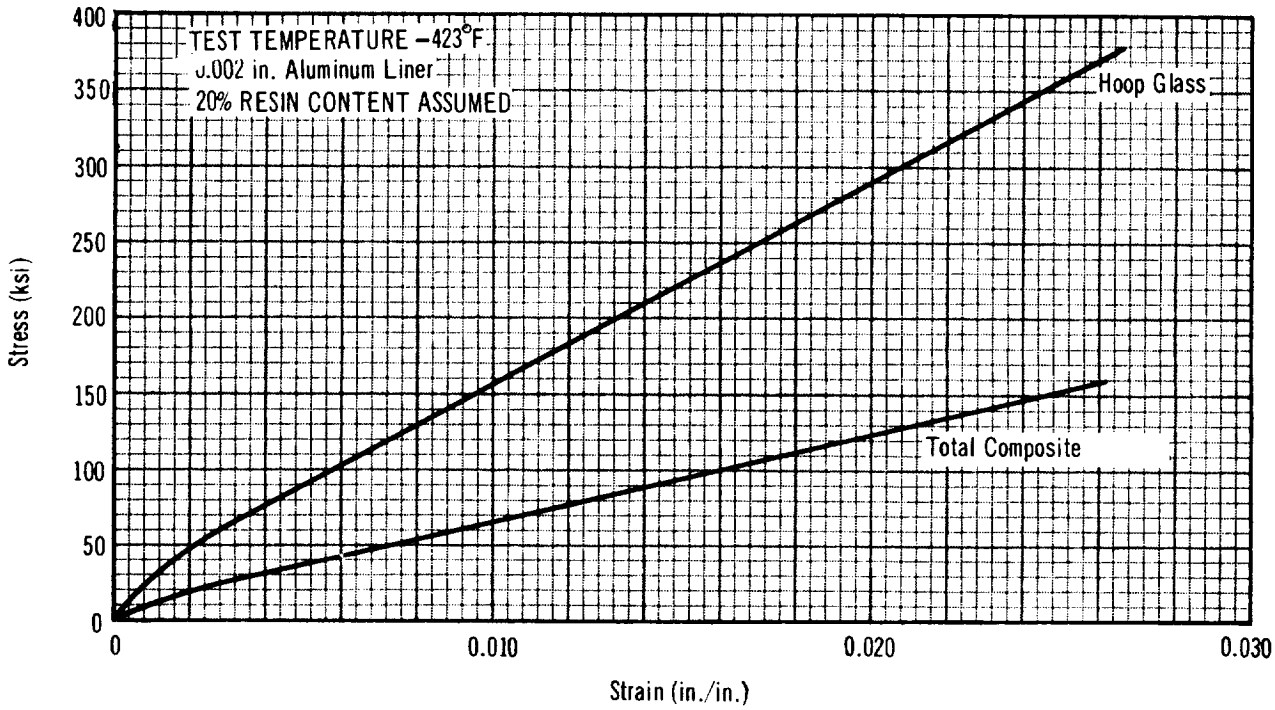


Figure 34. Vessel TA-1 Hoop Stress-Strain Diagram

The vessel remained relatively well-sealed throughout the test. No apparent leakage occurred across the NAFLEX seal.

The vessel failed on the initial intended cycle to 550 psi at 403 psi.

Post-test inspection of the vessel revealed that failure occurred in the longitudinal glass because of a displacement, caused by the hoop overwinding in the build-up area. The displacement was much more severe than noted during the fabrication. The vessel is shown post-test in figures 35 and 36. During the fabrication of the vessels from TA-3 onward special attention was paid to eliminating the displacement problem.

The primary liner remained satisfactorily bonded throughout the vessel.

Vessel TA-3: This vessel was cyclic tested at -423°F. End-seal configuration was of the type shown in figure 22b. The vessel was cycled to 2% strain at 550 psi for 101 cycles. This was followed by two attempted burst cycles. The maximum pressure attained on the burst attempts was 580 psi. Because of excessive leakage, the vessel could not be burst. The post-test leak check revealed three areas of leakage near the liner longitudinal seam. No leakage was detected at or near the bonded-on flange plate.

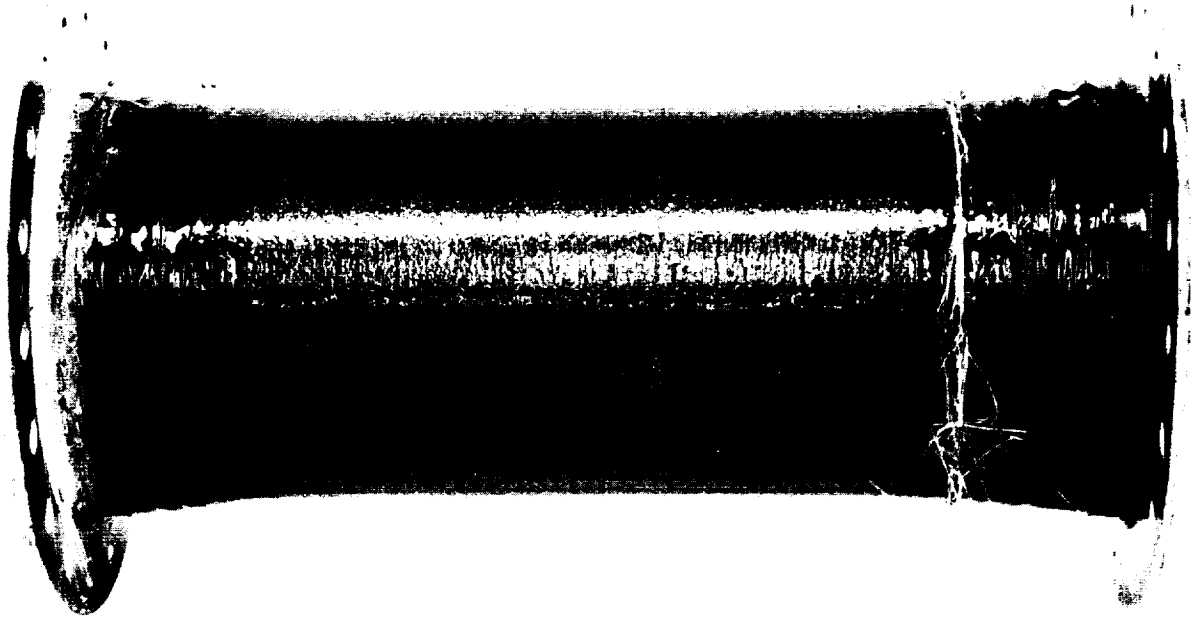


Figure 35. Vessel TA-2–Post-Test Overall View

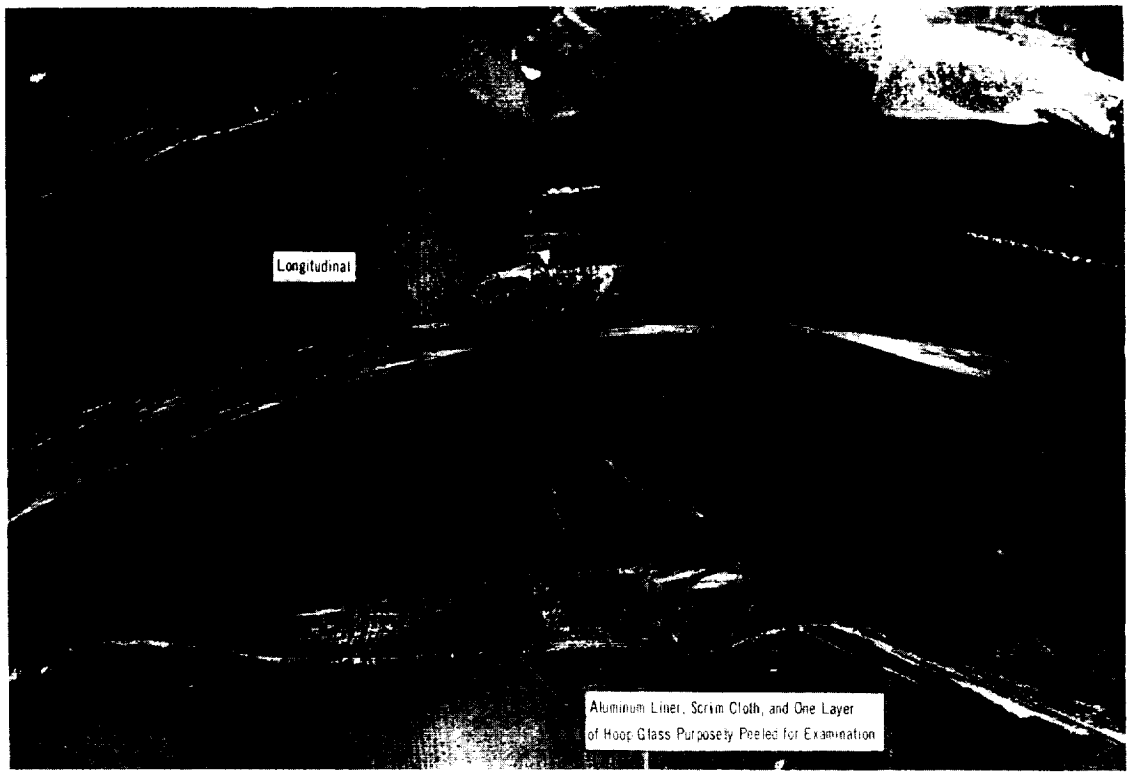


Figure 36. Vessel TA-2 – Post-Test Closeup View of Failure End

The exterior of the vessel appeared satisfactory, with only a minimum of resin crazing. The primary aluminum liner remained in excellent condition except for the longitudinal seam. There were no voids, bubbles, delaminations, wrinkles, or buckles in the primary liner test section.

High vacuum was lost in the chamber on the first cycle when the specimen pressure reached 400 psi, but some vacuum was maintained in the chamber through about 50 cycles which indicated only a very slight leak in the test specimen.

Vessel TA-4: This vessel was cyclic tested at -423°F. End seal configuration for this and all the remaining cryogenic work was of the type shown in figure 22c. The vessel was pressurized three times to 2% strain of 550 psi. Subsequent attempted pressurizations to 550 psi were unsuccessful. Leakage in the vessel was higher than the pump could overcome. Seventeen additional cycles were accomplished at declining pressures from 474 psi to 395 psi.

All vacuum was lost in the chamber during the first cycle. Initial leakage was indicated at 495 psi.

As with vessel TA-3, the primary aluminum liner remained in excellent condition except for the longitudinal doubler. The liner seam, however, was distorted and various leak paths through the seam were found. The doubler could easily be peeled from the liner seam and apparently served no useful purpose. Subsequent vessels contained no longitudinal doubler.

Vessel TA-5: This vessel was scheduled for a burst test at +75°F. The vessel failed at an internal pressure of 514 psi. Failure occurred at the start of the longitudinal reinforcement.

The complete liner including longitudinal seam remained in satisfactory condition.

Stress-strain curves for the vessel are shown in figure 37.

Vessel TA-6: This vessel was cyclic tested at +75°F. The vessel was pressurized 23 times to 2% strain at 500 psi. Testing was terminated at that point because of excessive liner leakage.

A leak was noted during the 19th cycle. Leakage was noted on the 20th cycle at 350 psi and increasing leakage occurred on each successive cycle.

Post-test examination revealed that the liner was debonded in a major portion of the test section and was wrinkled and cracked (fig. 38). No evidence of failure initiation was evident in the longitudinal seam.

Vessel TA-7: This vessel was cyclic tested at +75°F. The vessel was pressurized 41 times to 2% strain at 500 psi. Testing was terminated at that point because of excessive liner leakage.

A water leak into the chamber was noted at the end of the fifth cycle. Increased leakage was noted at the end of the 15th cycle and increasing leakage occurred on each successive cycle.

Post-test examination revealed the liner to be severely wrinkled and cracked (fig. 39).

Vessel TA-8: This vessel was burst tested at +75°F. The test was to verify the design and fabrication of a vessel without the short longitudinal reinforcement in the transition section (See Task II, fabrication of TA-8).

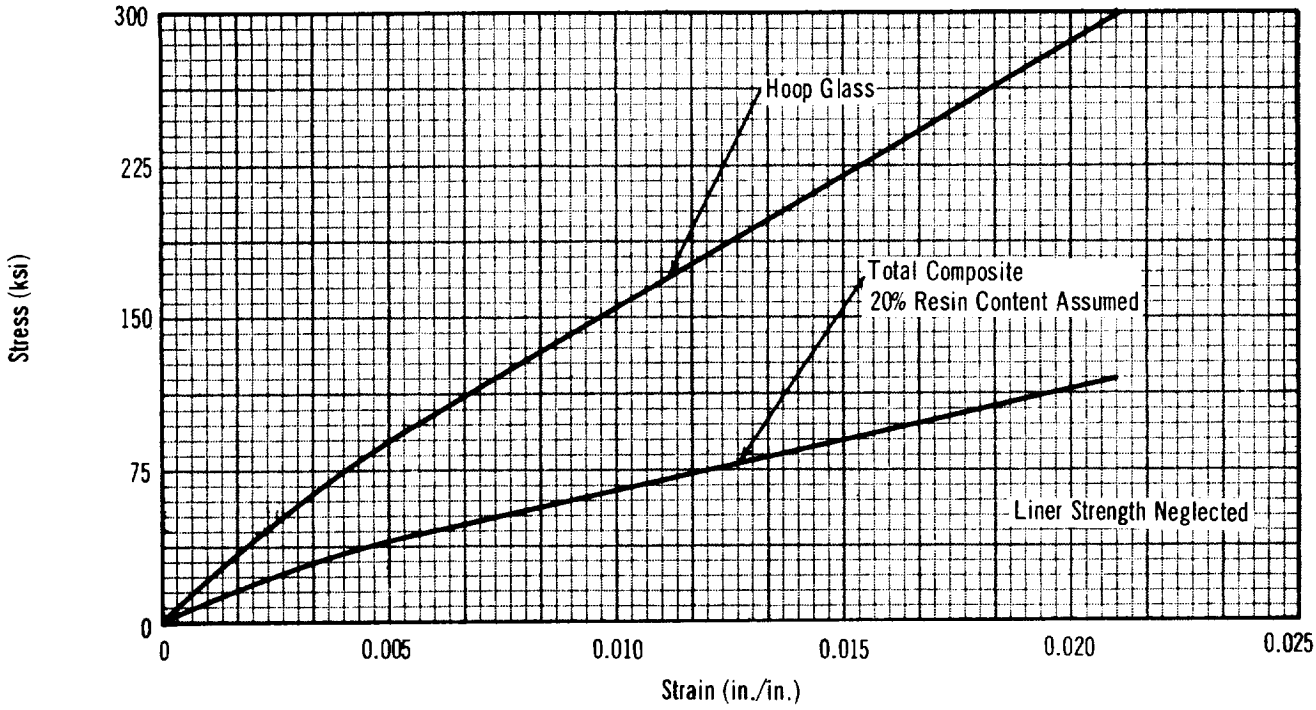
The vessel failed at a pressure of 630 psi. Failure occurred along a plane through the flange buildup at the longitudinal reinforcement (fig. 40). It was thought that the plane of weakness was caused by the interrupted cure of the vessel.

Vessel TA-9: This vessel was burst tested at +75°F.

Leakage was noted at 530 psi and the vessel failed at a pressure of 678 psi. Failure again occurred through the longitudinal reinforcement plane (fig. 41). The remaining vessels were fabricated with the longitudinals placed directly on the first hoop wrap layer. This placed the longitudinal reinforcement in single-shear rather than the previous (supposedly more advantageous), double-shear condition.

At points other than those of obvious structural shell failure, the liner remained in satisfactory condition.

Vessel TA-10: This vessel was burst tested at +75°F.



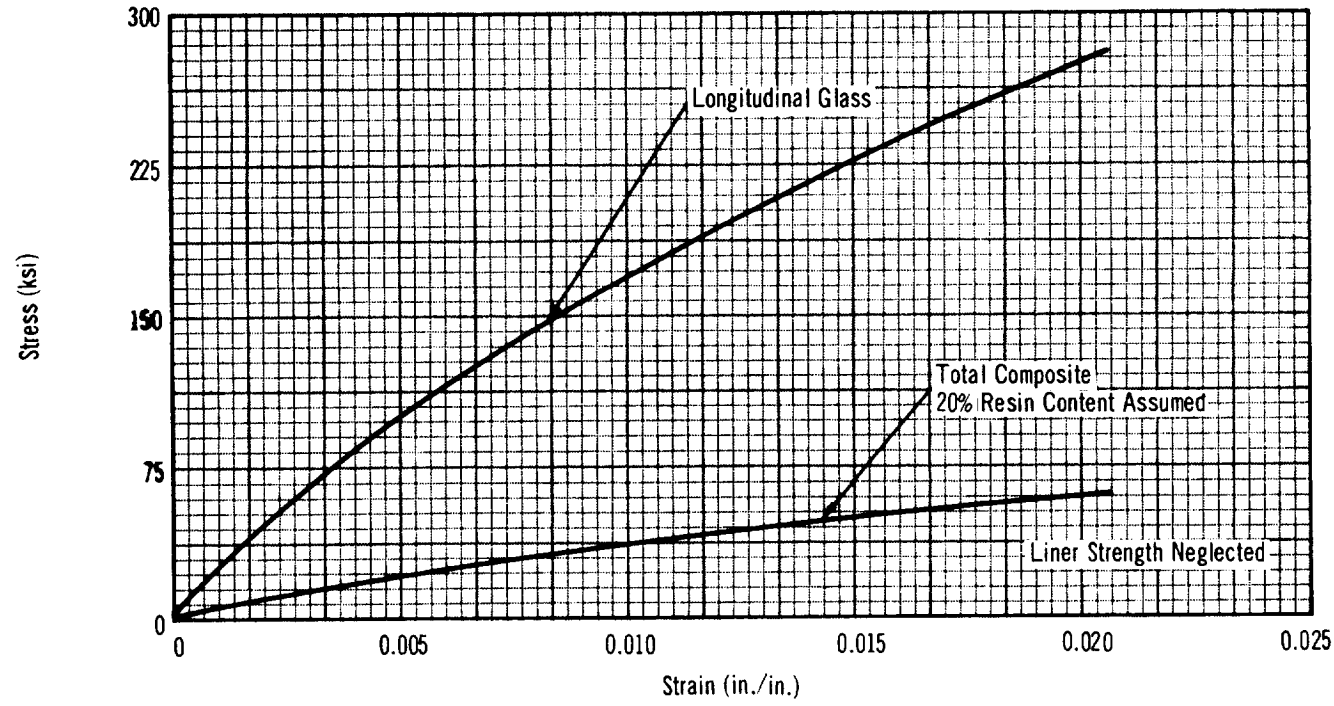


Figure 37. Vessel TA-5 – Stress-Strain Diagram Ambient Temperature Test

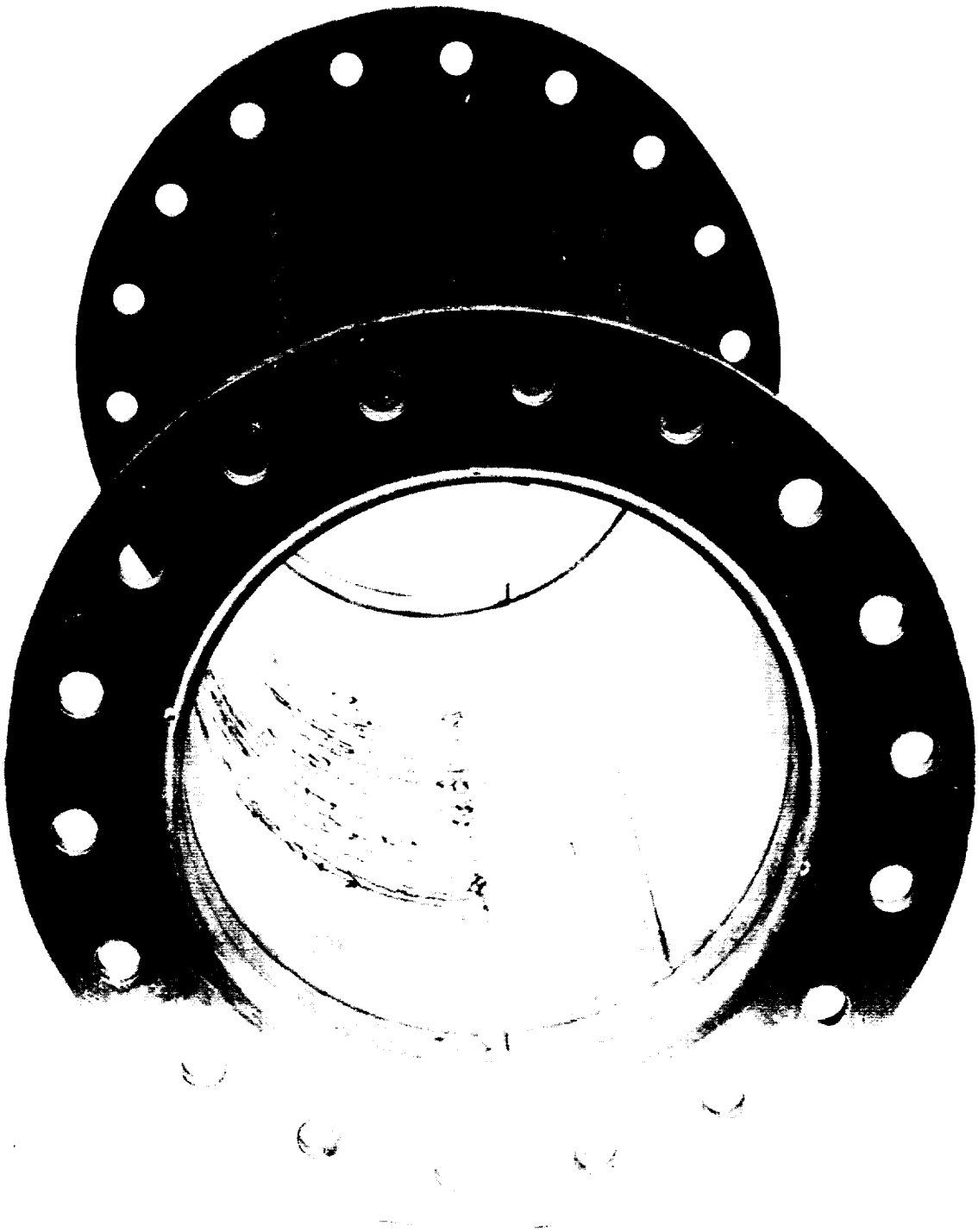


Figure 38 Vessel TA-6-Buckeled and Wrinkled Liner

Figure 39 Vessel TA-7–Post-Test, Interior

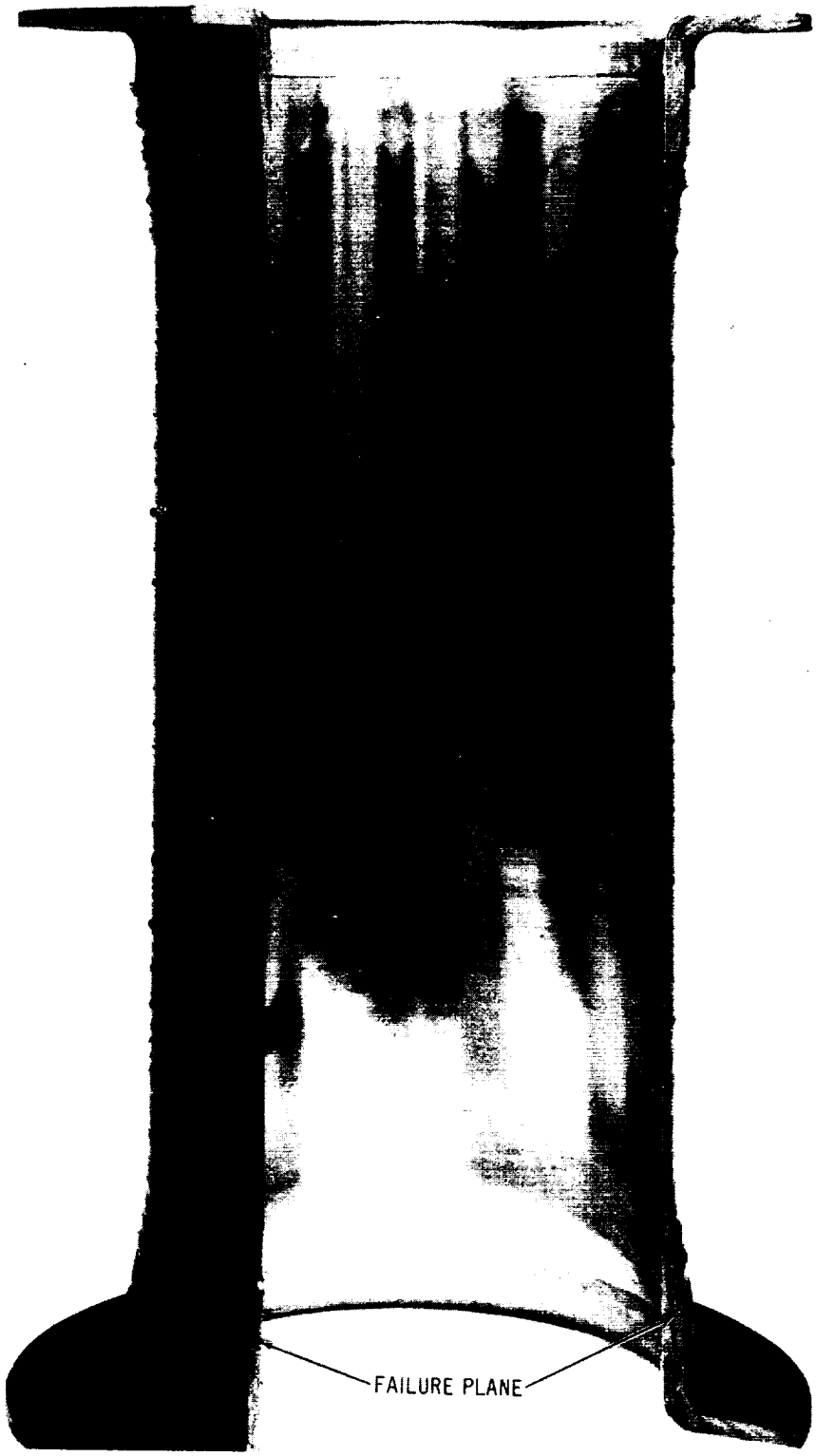


Figure 40 Vessel TA-5 Vessel Cross-Section

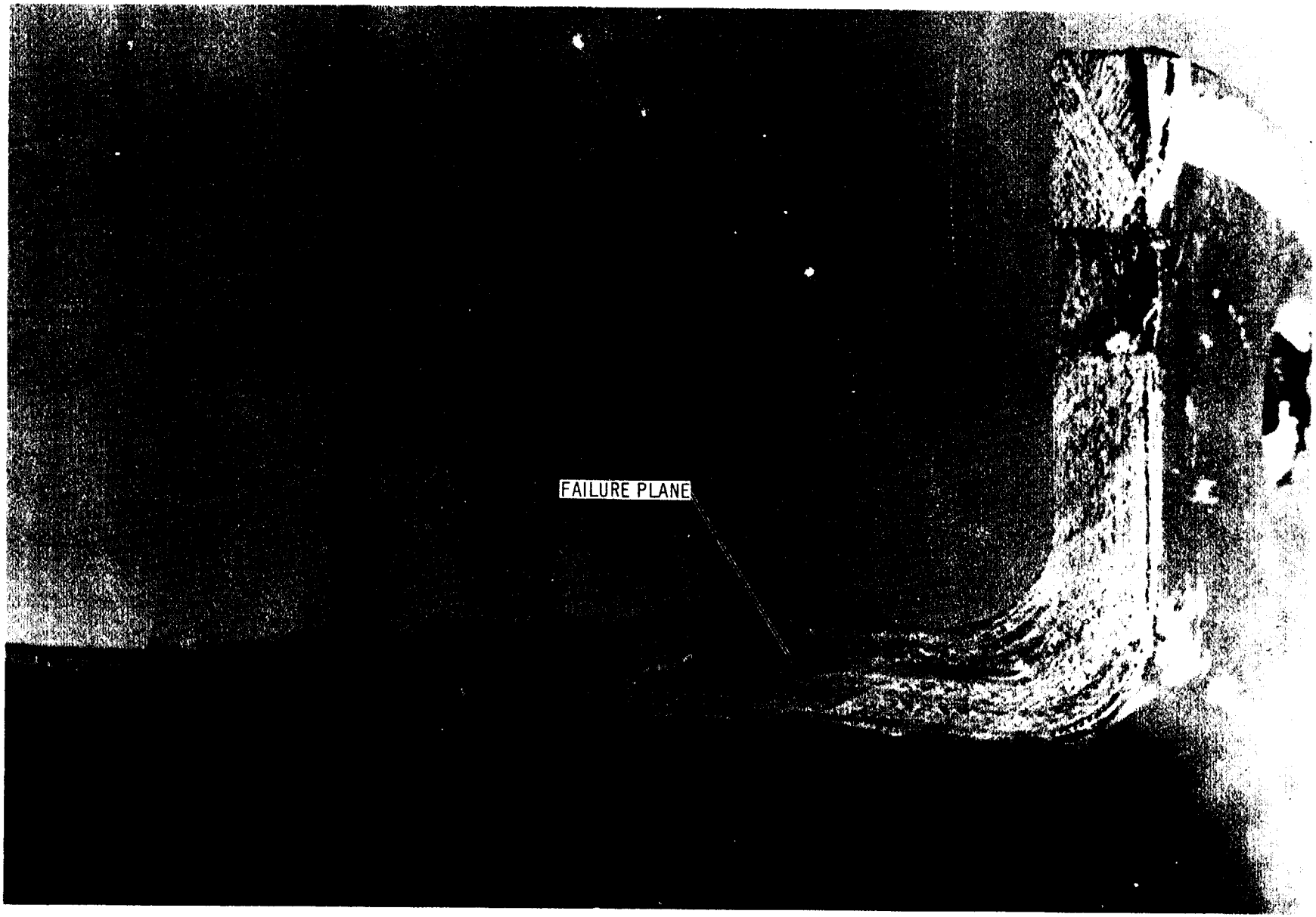


Figure 41. Vessel TA-9 – Close-up of Failure Cross-Section

Leakage was noted at 640 psi and the vessel failed at a pressure of 774 psi. This represented a total composite hoop stress of 192 300 psi, which approximated the expected strength of the S-glass at +75°F.

Vessel TA-11: This vessel had been fabricated with a nylon scrim in an attempt to increase the ambient temperature cyclic resistance of the liner-adhesive system.

This vessel was cyclic tested at +75°F. The vessel was pressurized 40 times to 2% strain at 500 psi. Testing was terminated at that point because of excessive liner leakage.

Leakage at one of the bolt holes was noted at the end of the first cycle. Other leakage into the chamber (which could not be pinpointed) was noted on the third cycle and increasing leakage occurred on each successive cycle.

Post-test examination revealed the liner to be severely deformed into a waffle pattern, which duplicated the underlying nylon scrim cloth (fig. 42). Leak checking of the vessel revealed many points of leakage. The liner, however, remained satisfactorily bonded to the structural shell, which was dissimilar to the liner failure mode of vessels TA-6 and TA-7. The nylon scrim did affect the ambient temperature cyclic resistance in the desired manner. The weave, however, of the cloth was such that the liner deformed severely between the nylon fiber strands.

Vessel TN-1: This nickel-lined vessel was burst tested at +75°F. The vessel failed at an internal pressure of 574 psi. Liner failure by buckling, debonding, and cracking occurred at the fiberglass build-up transition, the nickel deposition transition, and in the test section, (fig. 43).

Phase II Testing. - As stated earlier, the goal of the program was the development of a liner, which would withstand repeated cyclic loading, limited only by vessel failure, over a temperature range of +75°F to -423°F.

As indicated from the tests of vessels through TA-11 and TN-1, the 0.002-in.-thick aluminum foil worked satisfactorily (i.e., in areas of adhesive-bond retention, the liner successfully survived the excursions to high plastic strains without failure).

The initially selected adhesive system, Adiprene L-100:Epi-Rez 5101 (70:30) MOCA and the No. 112 fiber glass scrim cloth performed satisfactorily at cryogenic temperatures in the primary liner area (it appeared that leakage in all cases had occurred through the longitudinal seam). However, at +75° the liner buckled from the wall in large areas and otherwise deformed and failed as demonstrated in vessels TA-6, TA-7, and TN-1.

On this basis, it was deemed more desirable to rely upon the one type of metal liner (aluminum) and devote the remainder of the program to an

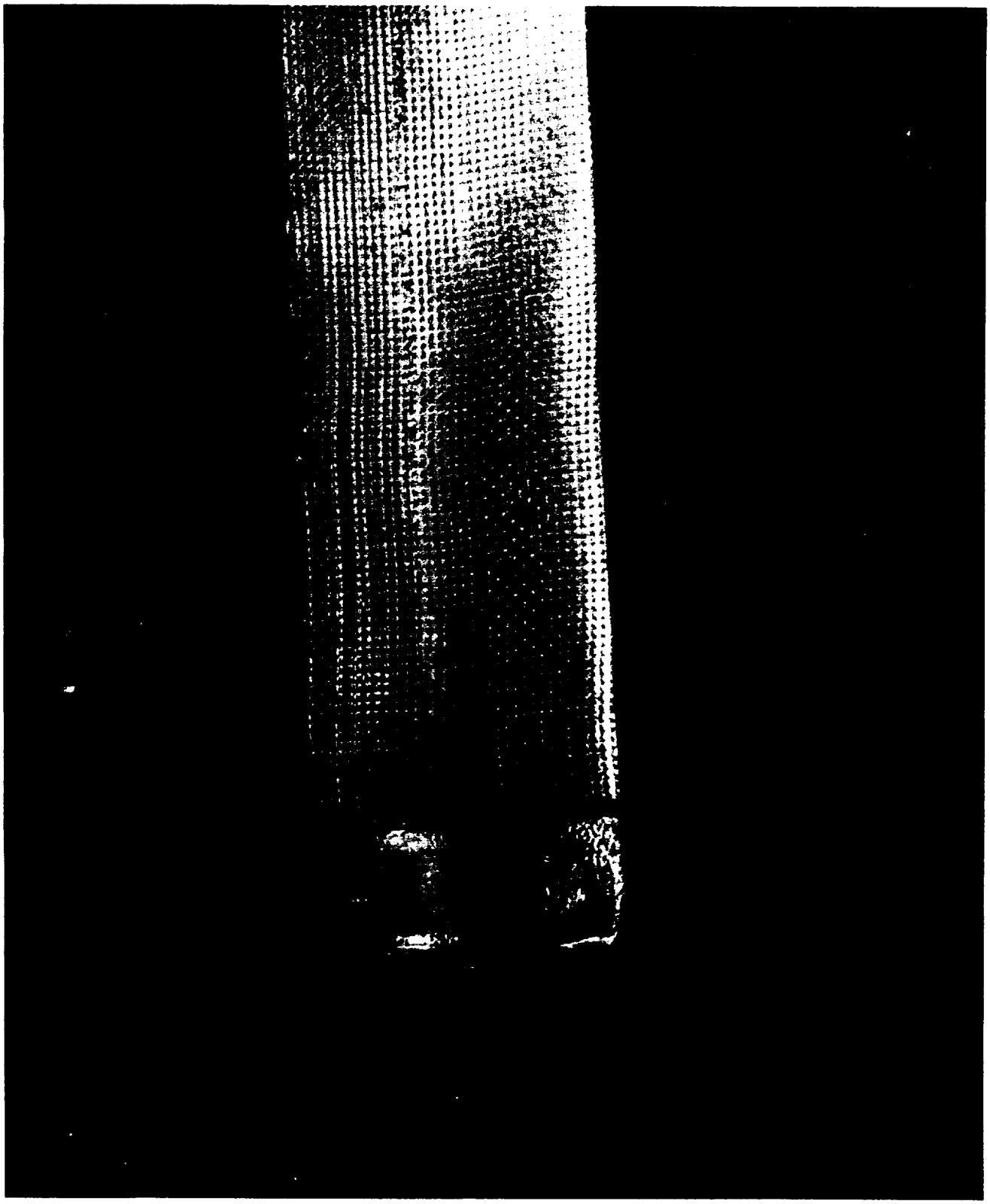


Figure 42. Vessel TA-11 – Waffle-Patterned Liner Post-Test

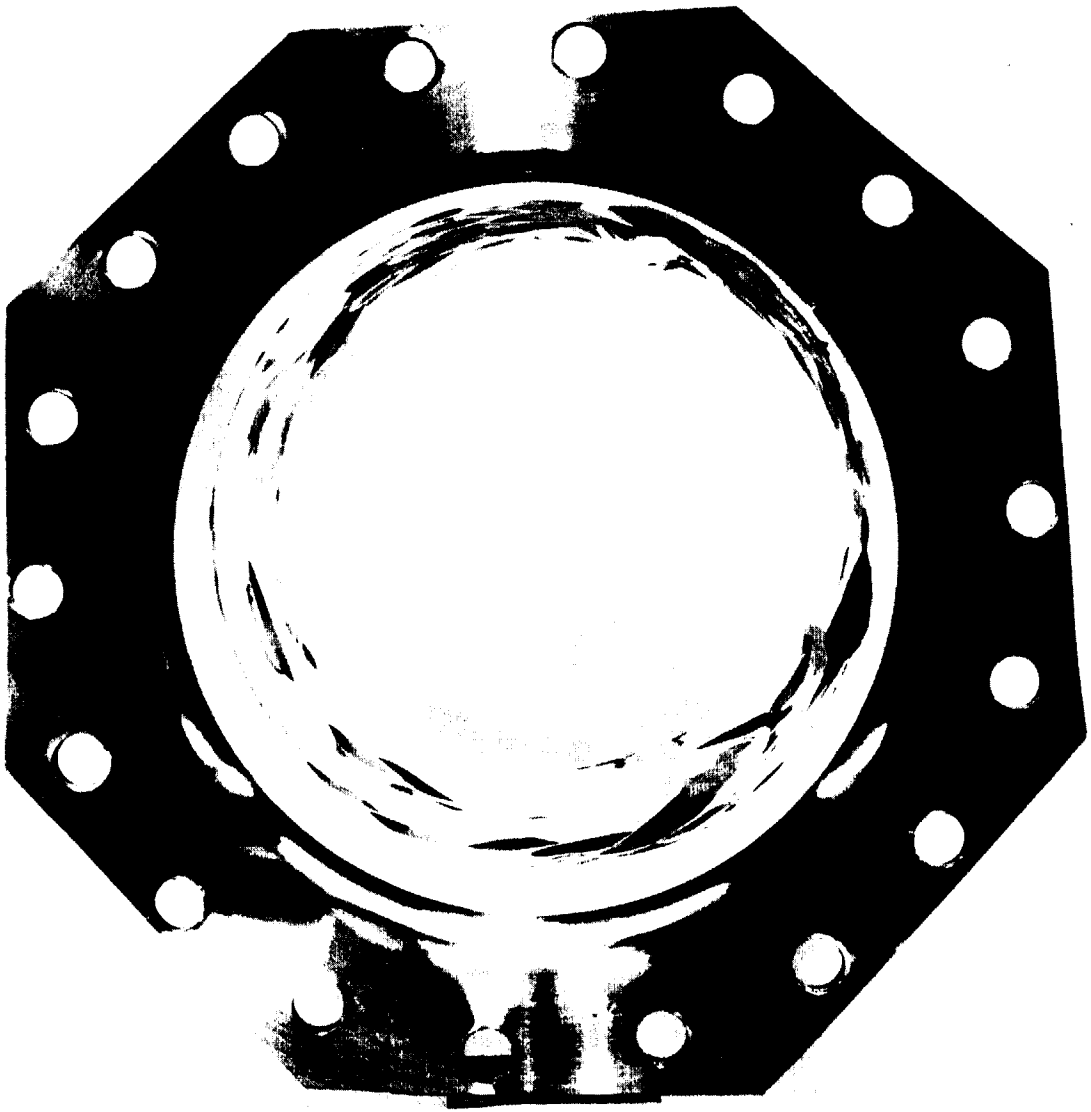


Figure 43. Vessel TN-1 – Post-Test Buckled Liner

evaluation of several other adhesive systems, rather than testing nickel liners with the 70:30 polyurethane/epoxy/glass system.

Nylon scrim cloth was used because of its apparent better tenacity at ambient temperature than the glass scrim cloth as exhibited in the test of vessel TA-11. However, a finer weave nylon was used to prevent the high liner deformation which occurred between the weave of the 2951/C material.

The test program was revised to reflect testing each vessel at both ambient and cryogenic temperature. First, 5 cycles were to be made at +75°F to 2% strain and following that, 10 cycles to 2% strain at -423°F.

Vessel TA-12: This vessel was cycled five times at ambient temperature from 0 to 500 psi. A helium leak check revealed no sign of leakage. The liner appeared to be satisfactory throughout the vessel.

The vessel was sealed and reinstalled in the chamber for the -423°F testing. During the initial pressurization to 550 psi, the vessel failed at 547 psi. Failure occurred in the longitudinal glass in the flange buildup. As mentioned in the Task II information, this vessel had the longitudinals running through the middle of the flange buildup. Also, a possible cause for failure was that the longitudinal glass had been in storage for an extended period (3 to 6 months) and deterioration may have occurred.

The appearance of the liner in areas beyond the points of vessel structural failure appeared good. No debonding or buckling was noted.

Vessel TA-13: This vessel was cycled two times at ambient temperature from 0 to 500 psi. During the third cycle, the vessel failed in the middle of the test section at 490 psi. Some water leakage had occurred during the first and second cycles.

The test section appeared satisfactory during the fabrication. As was the case for vessel TA-12, a possible explanation for the adverse vessel behavior was that the vessel had been fabricated with glass which had been stored for a considerable period.

Vessel TA-14: This vessel was cycled five times at ambient temperature from 0 to 500 psi. Water leakage occurred through the structural wall during each of the cycles. However, no debonding or buckling of the liner was noted in the examination. At the areas where leakage was noted on the vessel exterior, a pinhole or very fine crack was noted in the interior. The possibility of locating all such leaks and repairing them with an aluminum foil overlay was slim and, therefore, a coating technique was used. Two coats of G-207 were applied to the entire interior of the vessel as a sealant.

The vessel was cycled 11 times at -423°F. Pressures of 550 psi were achieved on all cycles except the 2nd, 10th, and 11th, which were 190, 538, and 453 psi, respectively. The low of 190 psi was apparently caused by a gas buildup in the lines; the last two cycles were low because of leakage through the liner seam. The primary liner remained in satisfactory condition. The longitudinal liner was deformed.

Vessel TA-15: This vessel was cycled five times at ambient temperature from 0 to 500 psi. No leak could be found with helium gas at 50 psi throughout the entire vessel. On removal of the test plates, no evidence of liner failure was found. The longitudinal seam was slightly deformed, but that was the only sign of distress.

The vessel was installed in the chamber for testing at -423°F and 10 cycles to 550 psi were achieved. A small vacuum (1 to 2 psi) was maintained in the chamber throughout the test, which indicated that the leak path was small because the driving force was 550 psi at maximum pressure. No evidence of liner failure was found, other than increased deformation of the longitudinal seam.

Vessel TA-16: This vessel was cycled five times at ambient temperature from 0 to 500 psi. No water leakage was noted on the vessel. No leakage was noted with helium gas at 50 psi. The liner appeared satisfactorily bonded to the wall throughout the vessel. Some slight deformation was evidenced in the longitudinal seam.

The vessel was installed in the chamber for testing at -423°F. Eight cycles to 550 psi were achieved (2% strain). Pressure on the 9th could be built-up to 495 psi and on the 10th cycle to 524 psi. There was no indicated leakage into the chamber during the first and second cycles as measured with the Pirani gage (pressure measured approximately 1 μ). The high vacuum was lost on the third cycle at approximately 290 psi and all vacuum in the chamber was lost during the seventh cycle.

The primary liner remained satisfactorily bonded to the wall. The seam was further deformed. Helium leakage could only be detected in the longitudinal seam area.

Vessel TA-17: This vessel was cycled five times at +75°F from 0 to 500 psi. No water leakage was evidenced during the test nor helium leakage during the post-test check at 50 psi. The primary liner remained satisfactory. The longitudinal seam was slightly deformed.

The vessel was installed in the vacuum chamber for testing at -423°F. Eleven cycles were achieved. Because gas apparently was trapped in the system, the first cycle only achieved 392 psi. Ten additional cycles were made to 550 psi.

The vacuum did not exceed 20 μ during the first cycle (392 psi) nor 155 μ during the second cycle (550 psi). During the 3rd cycle, the vacuum exceeded 2000 μ (limit of on-line monitoring gage), and all vacuum was lost during the 10th cycle.

The primary liner remained satisfactorily bonded to the wall. Helium leakage could only be detected in the seam area.

Vessel TA-18: This vessel was cycled five times at +75°F from 0 to 500 psi. No water leakage was evidenced during the test nor helium leakage during the post-test check at 50 psi. The primary liner remained satisfactory; the seam was very slightly deformed.

The vessel was installed in the chamber for testing at -423°F. Ten cycles to 550 psi were achieved.

The high vacuum was lost on the first cycle (i.e., pressure was greater than 2000 μ). However, pressure in the chamber did not exceed 2 psia throughout the test.

The primary liner remained in satisfactory condition. The seam remained in fairly good condition. Only slight leakage was evidenced during the helium post-test check.

Vessel TA-19: This vessel was cycled five times at +75°F from 0 to 500 psi. Water leakage was evidenced through the longitudinal seam during the fifth cycle. The primary liner remained in satisfactory condition.

The seam was sealed with two coats of G-207, similar to that for vessel TA-14.

The vessel was installed in the chamber for testing at -423°F. Seven cycles to 550 psi were achieved; cycles 8, 9, and 10 were to 470, 370, and 355 psi, respectively. All vacuum in the chamber was lost near the completion of the seventh cycle.

Several small areas appeared to have debonded in the primary liner. One such area leaked. The longitudinal seam was extensively deformed and leaked.

Vessel TA-20: This vessel was cycled five times at +75°F from 0 to 500 psi. No water leakage was evidenced during the test nor helium leakage during the post-test check at 50 psi. The primary liner remained satisfactory. The seam was deformed.

The vessel was installed in the chamber for testing at -423°F. The first cycle reached a maximum of 410 psi (gas had developed in the system). Seven additional cycles were to 550 psi; three additional cycles were to 470, 455, and 450 psi, respectively.

Leakage through the longitudinal seam was extensive. The seam was extensively deformed and the deformation extended into the primary liner (fig. 44).

Vessel TA-21: This vessel was cycled five times at +75°F from 0 to 500 psi. Water leakage was evidenced from the third cycle onward. Leakage had occurred at a point about 140° from the longitudinal seam. No leakage was noticed through the seam.

The area was sealed with G-207, similarly to that used for TA-14, except that the affected area was the only area treated.

The vessel was installed in the chamber for testing at -423°F. Six cycles were to 550 psi; four additional cycles were to 511, 437, 414, and 394 psi, respectively. All vacuum was lost on the second cycle.

Post-test examination of the vessel indicated a leak in the liner at the sealed leakage area. This is the area which had leaked during the ambient temperature test and had then been repaired.

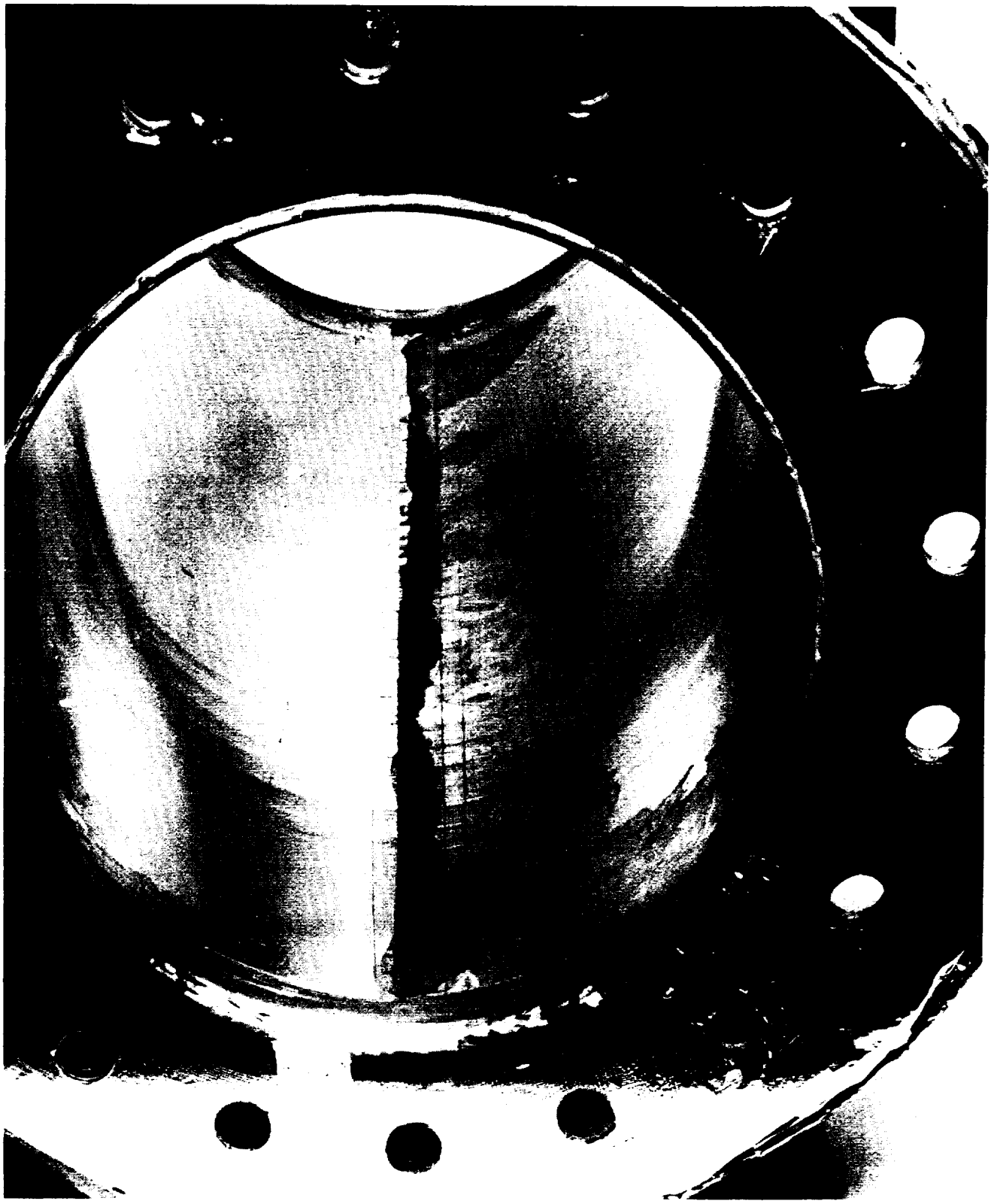


Figure 44 Vessel TA-20, Longitudinal Seam Deformation

SUMMARY OF RESULTS

Results obtained during the course of the program include the following:

(1) Toughness of various adhesives was measured by the drum-peel test at various temperatures including +75°F, -320°F, and -423°F. The adhesives systems tested were:

(A) Polyurethane (L-100), MOCA hardener, and the following:

1. Glass scrim cloth.
2. Nylon scrim cloth.

(B) Nylon - epoxy (FM 1025) (B-staged as received).

(C) Polyurethane (L-100): epoxy (Epi-Rez 5101) (70:30), MOCA hardener, and the following:

1. Glass scrim cloth.
2. Two nylon scrim cloths.

(D) Polyurethane (L-100): epoxy (Epi-Rez 5101) (80:20), MOCA hardener, and one nylon scrim cloth.

(E) Epoxy (Epi-Rez 5101), amine hardener (APCo 322), and the following:

1. Glass scrim cloth.
2. Nylon scrim cloth.

(F) Polyester (G-207) (vendor-supplied hardener) and the following:

1. No scrim cloth.
2. Epoxy-impregnated nylon scrim cloth.
3. Polyester-impregnated nylon scrim cloth.

(2) Lap-shear tests, uniaxial tensile tests, and thermal contraction tests were performed on several of the adhesives given above.

(3) Four adhesive systems were selected for incorporation into subscale pressure vessels. These four were as follows:

(A) (70:30) L-100:5101 with glass scrim.
(B) (70:30) L-100:5101 with nylon scrim.
(C) (80:20) L-100:5101 with nylon scrim.
(D) G-207 with no scrim.

(4) A pressure vessel was designed and fabricated to achieve a 1:1 strain ratio in the test section.

(5) Electrodeposited nickel was obtained which met the program specifications. Test vessel deficiencies did not permit a complete evaluation of the material and a program orientation eliminated the use of nickel liners.

(6) It was shown that thin (0.002-in. thick), soft (A 1100-0), aluminum foil could be used as a vessel liner, which was strained to 2% repeatedly, and the material would not fail.

(7) Vessels with bonded aluminum liners were cycled five times to 2% strain at ambient temperature with no leakage through the primary liner or seams.

(8) Vessels with bonded aluminum liners were repeatedly cycled to 2% strain at -423°F with no leakage through the primary liner. After a -423°F test, the longitudinal seam in all cases exhibited leakage.

(9) Problem areas worthy of further investigation are the following:

(A) Improved bond for the metal-metal liner seams.

(B) Consideration of other methods of thin-aluminum-liner fabrication (e.g., welding, electroforming, etc.).

(C) Evaluation of the thin-aluminum-liner and the high potential adhesive systems in compound curvature flight-weight type pressure vessels.

Douglas Aircraft Company, Inc.

Santa Monica, California, 4 May 1967

APPENDIX A

COUPON SPECIMEN PROCESSING

Tensile Lap Shear Specimen

Specimen configuration was as shown in figure 5 of this report. Each metal strip had a 1/4 in. hole drilled 3/4 in. from the top for pinhole loading. The surface preparation is given below.

Aluminum. - Aluminum surfaces were prepared as follows:

(1) Solvent wipe with methyl ethyl ketone (MEK).

(2) Etch 20 min. at 150°F in a solution of 15% sulfuric acid and 9% sodium dichromate.

(3) Rinse in distilled water and force-dry at 160°F for 20 min.

(4) Apply thin brush coat (approx. 0.0005-in. thick) of silane primer XD3901 to the bonding surfaces and allow to dry for a minimum of 30 min.

Electroformed Nickel. - Nickel surfaces were prepared as follows:

(1) Solvent wipe with MEK.

(2) Vapor hone with aluminum oxide.

(3) Rinse with distilled water and force dry at 160°F for 20 min.

(4) Apply thin brush coat (approx. 0.0005-in. thick) of silane primer XD3901 to the bonding surfaces and allow to dry for a minimum of 30 min.

Bonding. - The test specimens were prepared as described in the following paragraphs. The etched specimen is placed in a bonding fixture. The mixing ratios for the adhesives were as follows:

(1) Adiprene L-100/MOCA--100 parts/11 parts.

(2) Adiprene L-100:Epi-Rez 5101/MOCA--70 parts:30 parts/19 parts.

(3) FM 1025--Preimpregnated B-stage film.

(4) Epi-Rez 5101/APCo 322--100 parts/15 parts.

The adhesives are throughly mixed and then degassed to remove any air and moisture.

The adhesive is applied to all bonding surfaces. One ply of the chosen scrim cloth is placed in the bonding area and wet completely with the adhesive.

Four, 1/2-in. wide strips of the Epi-Rez 5101/APCo 322 preimpregnated tape are applied to the bonding area in a direction parallel with the length of the adherend.

The adherends are assembled in the bonding fixture with a 3/4-in. overlap, loaded to 40-psi overpressure, and cured as follows:

(1) Polyurethane adhesive (Adiprene L-100/MOCA) under the following conditions:

(A) 72 hr at 77 $\pm$ 5°F with 5 to 7 psi.

(B) 24 hr at 160°F with 5 to 7 psi.

(2) Polyurethane - Epoxy adhesive (Adiprene L-100:Epi-Rez 5101/MOCA)

Same cure cycle as for the Adiprene L-100/MOCA.

(3) Nylon-epoxy adhesive (FM 1025), under the following conditions:

(A) 72 hr at 77 $\pm$ 5°F with 10 psi.

(B) 24 hr at 160°F with 10 psi.

(C) 90 min. at 250°F with 10 psi.

(4) Epoxy adhesive: (Epi-Rez 5101/APCo 322), under the following conditions:

(A) 72 hr at 77 $\pm$ 5°F with 5 to 7 psi.

(B) 24 hr at 160°F with 5 to 7 psi.

After the cure is completed the specimens are removed from the test fixture and are ready for testing.

Drum-Peel Specimens

Specimen configuration was as shown in figure 8 of this report.

To wind the fiberglass composite on the specimen, 1/2-in.-wide, 80 end SCG150 HTS 1/0 collimated, preimpregnated tape is used. The tape is the same composition and construction as that which is used for fabrication of the 7-1/2-in. diam test vessels.

The surface of the ring is treated in the same manner as in preparation of lap shear specimens described previously.

The mandrel is mounted in the NOL ring winding machine and held in place by the centering shaft. Collimated tape is mounted in the tensioner and tension adjusted to 40 lb (1/2 lb/end).

For liquid adhesive systems, 1/2-in.-wide scrim cloth strips are cut to a length equivalent to the circumference of the metal specimen rings. Adhesive ingredients are mixed and the scrim strips are impregnated. The first ring from one end flange is masked off 1 in. along the circumference and a thin adhesive layer is applied around the ring to wet the surface. The 1-in.-long masked area is uncovered and a 1/2-in.-wide, 1-in.-long, teflon, film strip is placed on the masked area. An impregnated scrim strip is wound around the ring circumference starting at one edge of the teflon separator strip. A butt joint of the scrim strip is thus made at one edge of the teflon strip.

The 1/2-in.-wide fiberglass tape is tied down around one end flange. Tape is wound over the ridge made by the first width restraining ring at the teflon strip position. The tape is positioned in the center of the specimen ring and the fiberglass composite is wound. The fiberglass tape is wound over each, successive-width, restraining ring after completion of the fiberglass winding on the preceding specimen ring and preparation of the succeeding ring. A final tie-down of tape is made at the second end flange.

The FM1025 system is a preimpregnated scrim cloth. In preparing specimens with this system, 1/2-in. strips wide are cut to a length equivalent to the circumference of the specimen rings. The teflon separator strip is placed on the metal ring and the strip of adhesive film is wrapped around the ring. The fiberglass tape is then wound around the adhesive to secure it against the metal ring and tape winding is continued until the desired fiberglass composite thickness is attained. Positioning of the teflon strip, the adhesive butt joint, and the beginning of fiberglass wrap is identical to that described for the liquid adhesive systems.

Second Series Drum-Peel Specimen

All processes were the same as previously discussed in the first series-except that mix ratio for the 80:20 L-100:5101 system was 80 parts L-100, 20 parts 5101, and 17 parts (by weight) of MOCA.

The cure cycle for the L-100:5101 was as follows:

(1) 16 hr at room temperature (70°F).
(2) 1 hr at 150°F.
(3) 8 hr at 250°F.

Specimens are cured on the mandrel at the desired conditions (see lap shear specimen preparation). After cure, the tape wound over each width restraining ring is cut and the specimens are separated from the rest of the mandrel. A sawcut is made in each specimen through the fiberglass composite above the butt joint of the adhesive to the teflon separator strip to provide a short tab which can be peeled back and gripped by the jaws of the testing apparatus. The specimens are then ready for testing.

Uniaxial-Tensile Specimen And Thermal Contraction Specimen

Specimen configuration for the tensile specimen was as shown in figure 12 of this report. Mix ratios for the resins were the same as given in the procedure for the lap-shear specimen.

Preparation was as follows:

(1) Heat L-100 resin to 200°F; heat fabric to 200°F and hold each material at temperature for 20 min.

(2) Degas L-100 and put in 120°F oven.

(3) Heat 5101 resin (where applicable) to 120°F.

(4) Place fabric in desiccater.

(5) Mix resin/resins with MOCA and degas the mixture.

(6) Fabricate laminate by completely wetting each ply of material with the resin mixture (25 plies of A1100 glass scrim; 10 plies of nylon scrim) and lay plies upon each other.

(7) Apply vacuum of 10-15 in. of mercury and set for 16 hr at 75°F.

(8) Place laminate in press with 40-psi pressure and pull vacuum and cure for the required period (see lap-shear specimen preparation).

Goodyear G-207 Drum-Peel Test Specimen

Specimen fabrication was similar to that of the drum-peel specimens above, with the following modifications.

The mixing ratio is 100 parts G-207B, 63 parts toluene, 27 parts MEK, and 4 parts G-207C.

The mixture is applied to the etched and primed ring in four brush coats with a 10-min. drying period between each coat.

The next steps in the process were as follows:

(1) For the case where no scrim is used, collimated tape is wound over the hardened G-207.

(2) For G-207-impregnated scrim, the scrim is applied to the G-207 coated ring and the scrim is then impregnated with G-207 until saturated (as much as possible).

(3) For epoxy-impregnated scrim, the scrim is impregnated with the wet resin and applied directly to the G-207 coated ring.

The collimated tape is then wrapped over the prepared mandrel in the same manner as stated previously.

Goodyear G-207 Uniaxial-Tensile Specimen And Thermal Contraction Specimen

Because of the high volatile content of the adhesive resin, the fabrication of laminates was difficult. The following procedure was used:

(1) Impregnate 12 sheets of glass cloth with the mixed adhesive (100/4, see G-207 drum-peel test specimen processing).

(2) Air-dry the sheets and then heat at 250°F for 1 hr to drive off additional volatiles. Repeat the impregnation and drying process until the sheets appear to contain an excess amount of resin.

(3) Combine the 12 sheets into 3 sets of 4 sheets and repeat the impregnation and drying process.

(4) Combine the three sets into one laminate.

(5) Place the laminate in a mold and heat to 200°F under a vacuum of 20-in. of mercury.

(6) Impregnate the laminate and heat to 300°F and pressure to 1000 psi for 2 hr. Repeat this impregnation, heating, and pressurization until the laminate is suitable for use.

Specimens fabricated in this manner will be approximately 0.090 in. thick.

APPENDIX B

205°F CURE STUDIES OF AN EPOXY RESIN AND EPOXY-POLYURETHANE BLENDS USING THE VIBRATING REED APPARATUS

Processing And Test Procedure

Mixtures of Epi-Rez 5101 resin, Adiprene L-100 resin and stoichiometrically equivalent amounts of curing agent for each resin (table B-I) were cast into thin films and precured until gelled. The gelled films were then removed from the backing sheets and vibrating reed specimens prepared. The gross physical condition (surface tack, elasticity, toughness, etc.) were noted at that time.

TABLE B-I

RESIN AND HARDENER RATIOS

Weight percent Epi-Rez 5101 resin	Weight percent L-100	Parts curing agent per 100 part blend
100	--	15[a]
30	70	19[b]
20	80	17[b]

[a] APCo 322
[b] MOCA

The specimens were then inserted in the test clamps in a 68°F oven and their room temperature frequency response was noted. The oven was brought to the cure temperature of 250°F and the cure started. The frequency response characteristics of the specimens were noted periodically during the cure cycle. The cycle was considered complete when the response characteristics became constant in resonance frequency and band width. The specimens were allowed to cool to room temperature and the room temperature response of the cooled specimens was then determined.

Results

The results of two cure cycles on each material are reported. The handling characteristics of the specimens before curing are as follows. In both runs, the 100% Epi-Rez 5110 appeared fully cured. It was hard, resilient, and brittle. The sheet sample of the 80:20 L-100:5101 blend was much softer than the epoxy, as would be expected, and was highly damped. However, it could be removed from the backing plate without excessive distortion. The 70:30 blend, on the other hand, was softer than the 80:20 blend, and had a soft "tacky" surface. The cast sheet required a longer

cure [1 to 1-1/2 hours at 250°F] before it could be removed from its backing sheet without excessive distortion of the sheet.

The moduli and half width damping factor from the first run are reported in figures B-1 through B-3. The data from the second run are reported in figures B-4 through B-5.

The second-run data were generated to verify the cure times established in the first runs, and to determine the effect of room temperature aging on the cure times found in the first cure runs. The data are reported separately because excessive clamping pressure inadvertently applied to the blend specimens caused a necking in the specimens at the clamp. In addition, the length marks on the 100% epoxy specimen were not discernible; and, therefore, the free length of the specimen could not be determined accurately.

The Young's modulus data were generated from the resonance frequency data, and the loss modulus was generated by making the necessary approximations as to specimen thickness and length required to establish the changes in values. Although the data accurately provide a measure of the cure time, they do not provide absolute values of modulus and loss modulus. Therefore, the moduli scales are arbitrary and illustrate the time relationships of the shape of the modulus and loss modulus cures.

The following paragraphs contain the detailed evaluation of each material.

100% Epi-Rez 5101 Resin. - The resin appeared cured after the initial oven cure. However, the Young's modulus and damping factor in the first run (fig. B-1) showed an initial decrease indicating that the material softened. This was followed by an increase in modulus and a decrease in the damping factor indicating that further cross-linking was occurring. The cure appeared to be complete in approximately 8-1/2 hr, as evidenced by the constancy of the modulus values.

The room temperature values of the Young's modulus, loss modulus, and damping factor were all monotonically increased indicating that the resin had undergone further cure; however, the changes were slight.

The second cure on fresh resin, unlike the blends, behaved differently from the first cure in that the Young's modulus remained constant after the initial drop because of the temperature increase to 250°F. The loss modulus, on the other hand, generally showed the same reaction pattern as in the first run. Analysis of the data indicates that the reaction causing the changes in loss modulus was essentially complete in approximately 9 hr.

80:20 (L-100:5101) Blend. - The reaction between the resins and the curing agent appears to occur in three steps. The first step, an increase in Young's modulus, is accompanied by an increase in damping and loss modulus. These latter values reach a peak just before the inflection

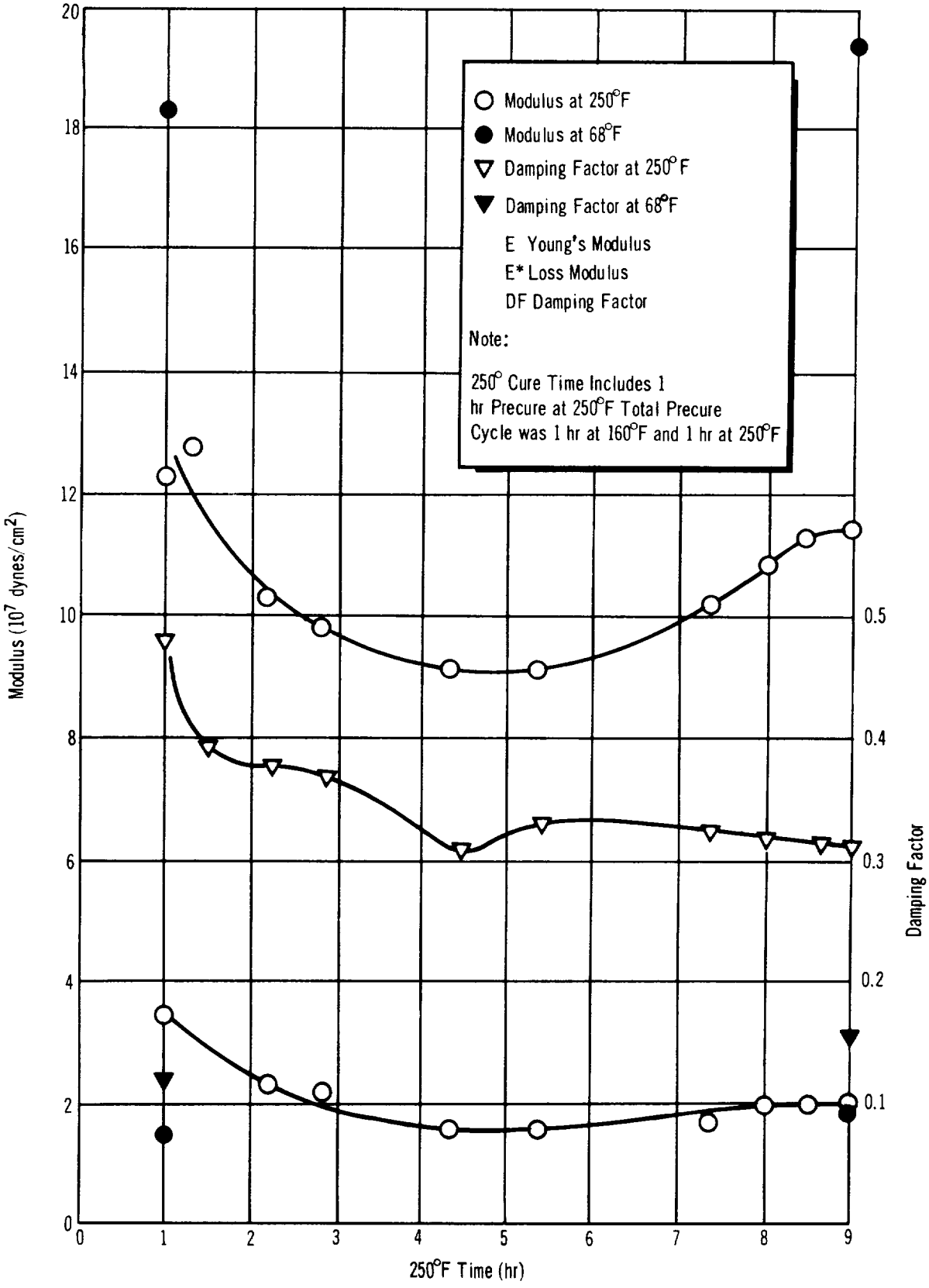


Figure B1. Cure Cycle 5101 Epoxy Resin

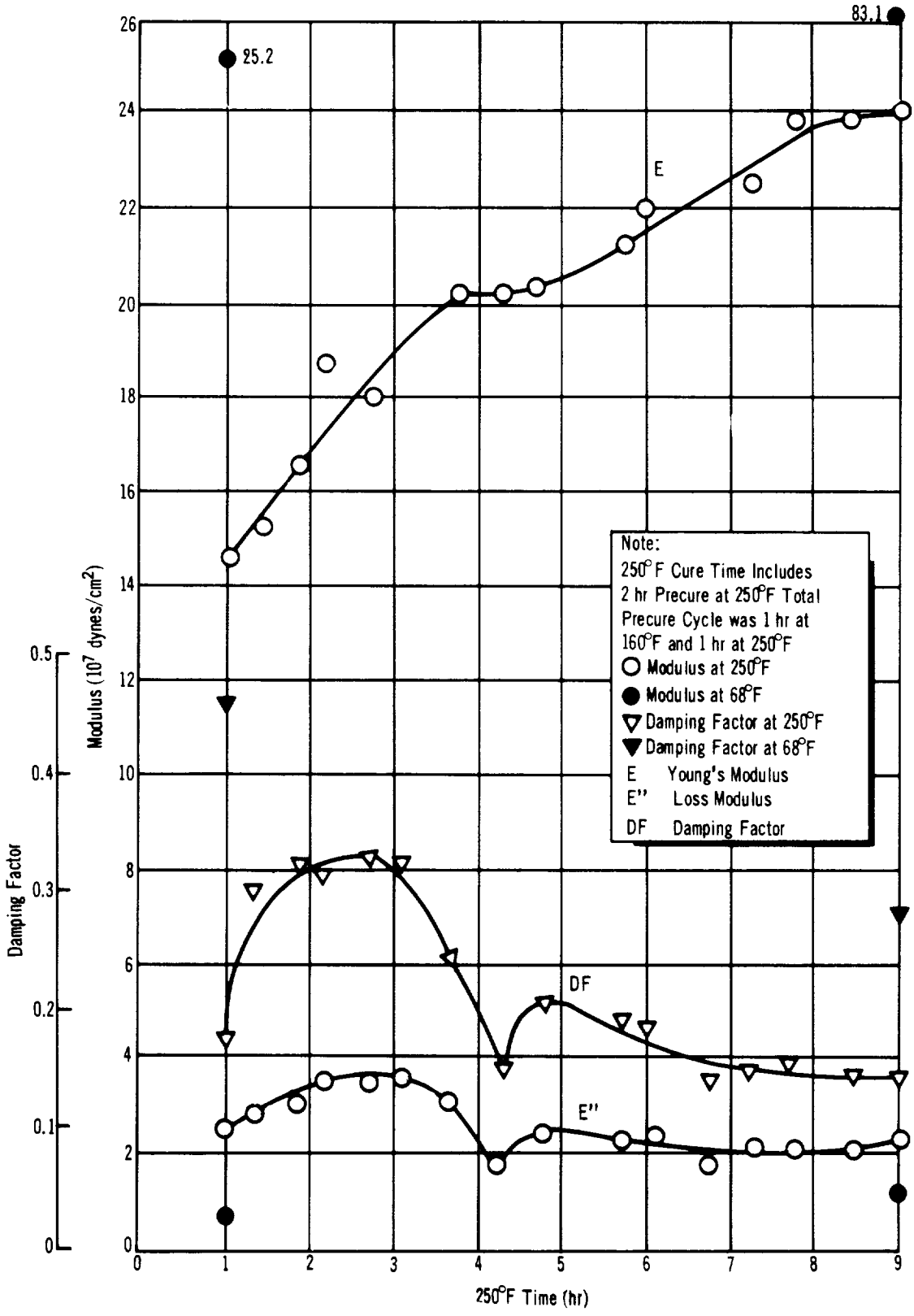


Figure B2. Cure Cycle 80% Polyurethane 20% Epoxy

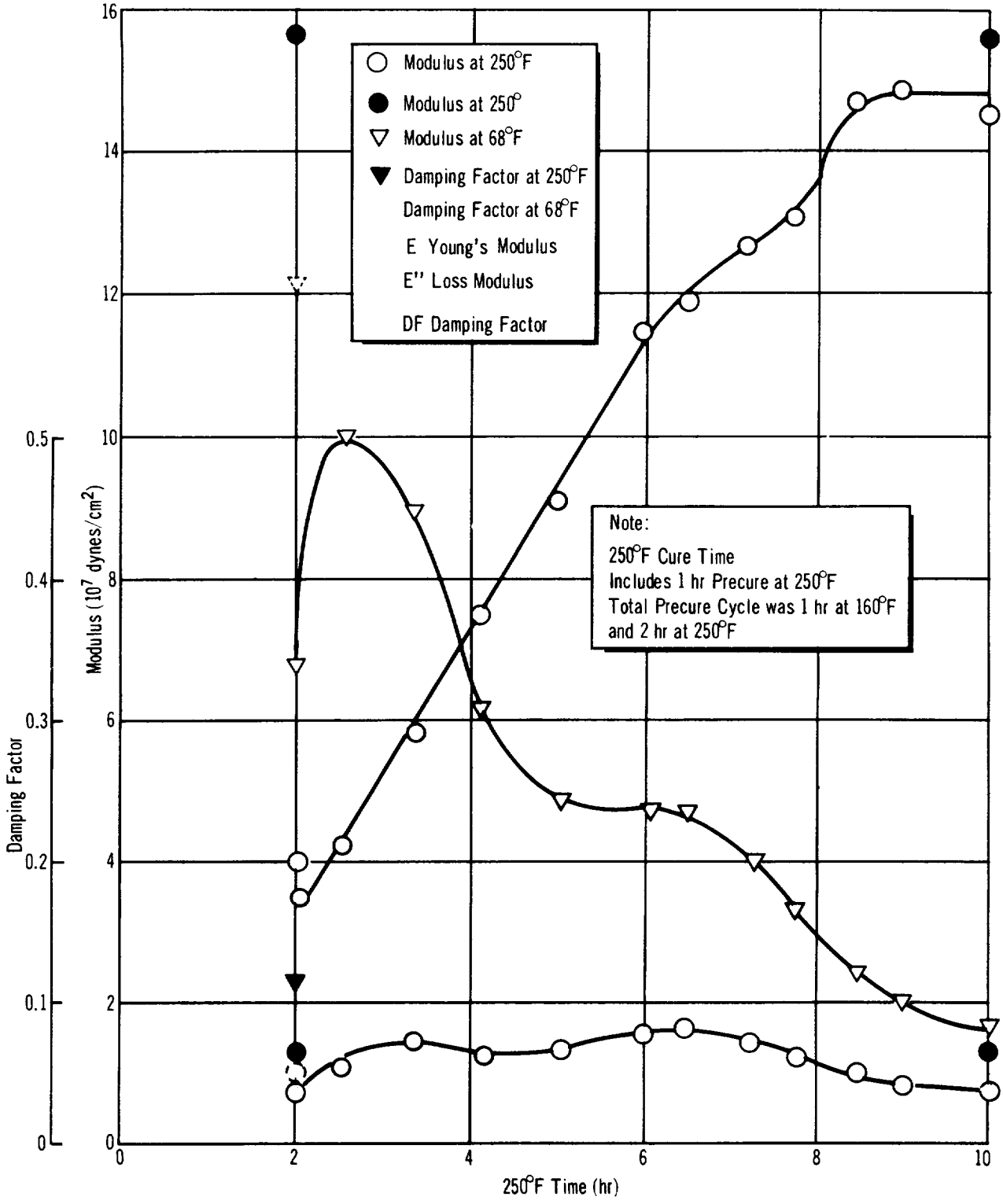


Figure B3. Cure Cycle 70% Polyurethane – 30% Epoxy

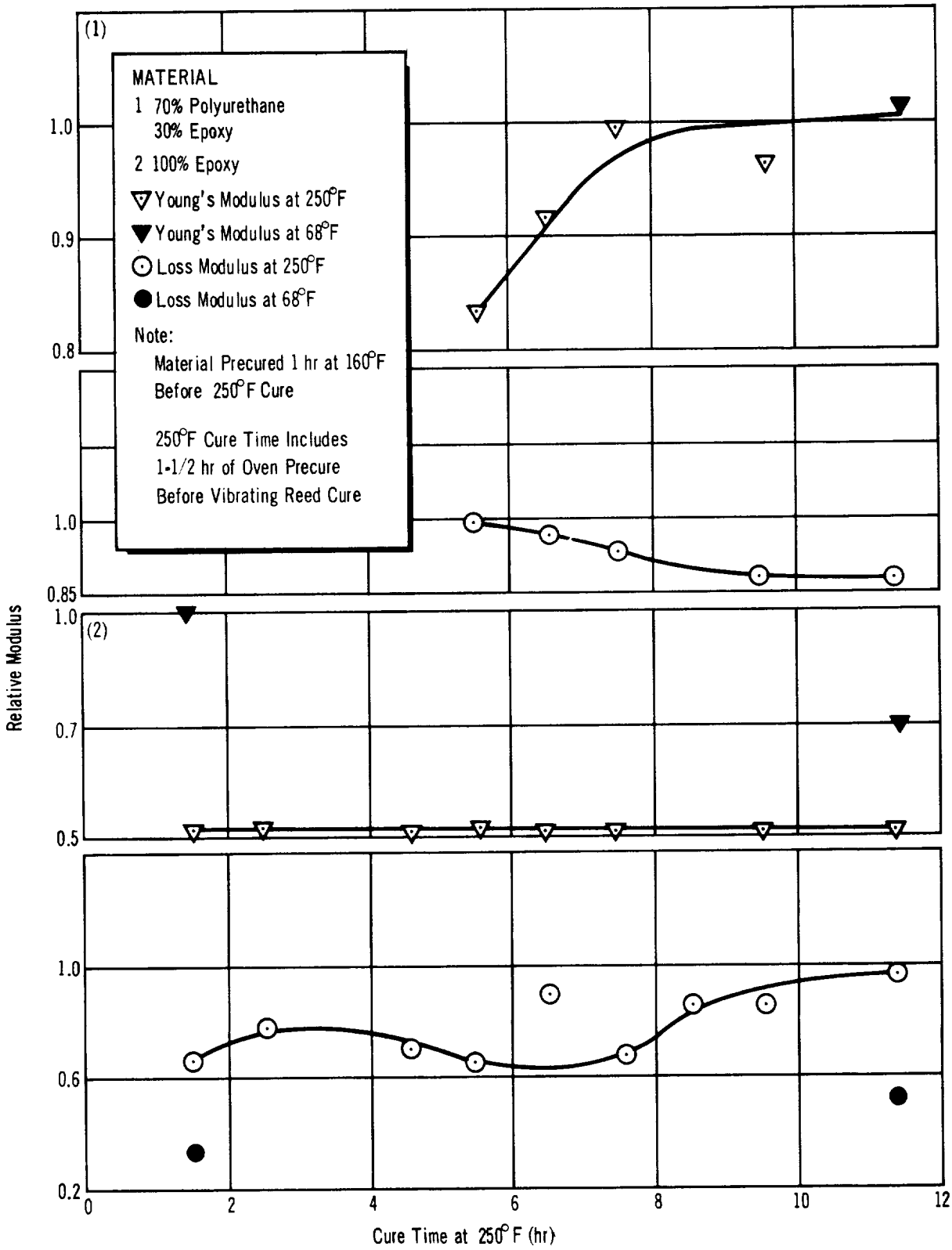


Figure B-4. Relative Modulus vs Cure Time Polyurethane Epoxy (70:30) Blend and 100% Epoxy

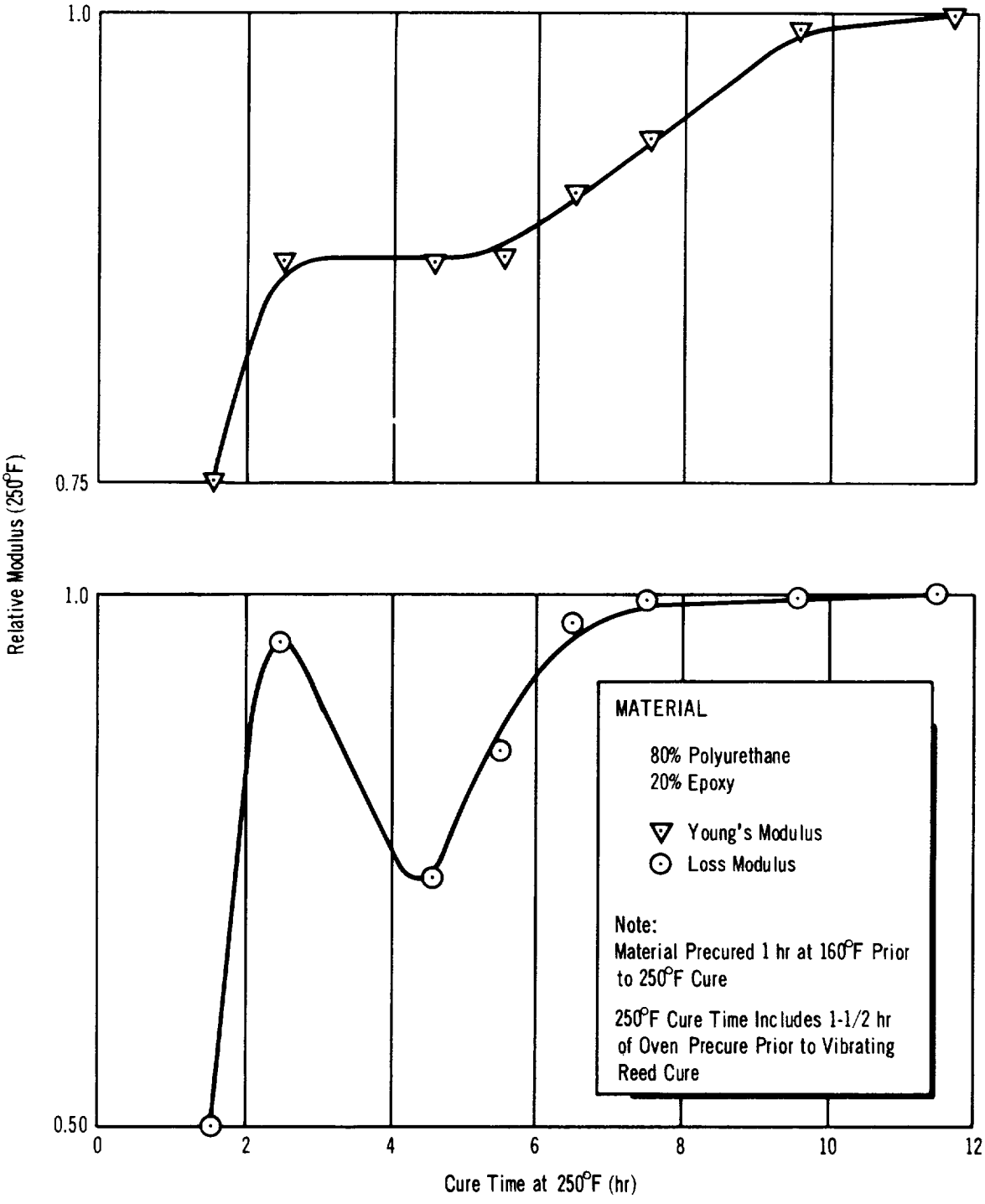


Figure B5. Relative Modulus vs Cure Time Polyurethane: Epoxy (80:20) Blend

point of the Young's modulus curve. The Young's modulus remains unchanged while the loss modulus and damping factor drop to a minimum value just before the inflection point in the Young's modulus curve. The second increase in modulus is again accompanied by a convex inflection of the damping factor and loss modulus in the first run, and an increase to a maximum value in the second run. All maxima occur shortly after the second inflection point in the Young's modulus. The fresh material, which had not been subjected to room temperature exposure, showed a much more rapid initial step followed by a longer "plateau period" in the Young's modulus curve. Examination of the inflection points would indicate that the reaction occurs in two steps, with approximately 9-1/2 hr required to complete both steps in the fresh material and only 8-1/2 hr to complete the same steps in the material which had received the room temperature aging. The degree of reaction is indicated by the sharp increase in the room temperature values for the Young's modulus and the decrease in damping factor after the cure. The loss modulus shows that the absolute energy-absorption capacity of the blend remains essentially constant because of the countereffects of the changes in the damping factor Young's modulus.

70:30 (L-100:1501). - The 250°F data follow approximately the same pattern in the first run as the 80:20 blend. However, the "plateau period" in the Young's modulus curve was only an inflection point, and the second "peak" in the damping factor curve disappeared, to be replaced by a plateau period. The modulus changes appear to be over after 9 hr, but analysis of the data shows the damping factor to be dropping slightly, which would indicate the cure reaction was still incomplete. The second run indicated that the moduli changes had stopped after approximately 9 hr, and then remained constant for 2-1/2 hr which confirmed the 9 hr cure period. The "hinging" effect, because of specimen compression, was so severe in this case, that frequency response data could not be obtained until the specimen had received a 4-hour cure in the vibrating reed apparatus.

APPENDIX C

VESSEL PROCESSING AND TEST FLANGE BONDING

The following paragraphs describe vessel processing and test flange bonding. Note that all procedures are numbered for ease of reference.

(1) Annealing And Flange Liner Forming

(1.1) Cut out 6 to 12 squares, 14-in. ± 1-in. by 14-in. ± 1-in., of 0.006-in. -thick A1100 aluminum foil.

CAUTION: Exercise extreme care to prevent damage to foil components by wrinkling, indenting, scratching, tearing, etc. Always wipe off contacting surfaces when positioning foil. If the foil is damaged, prepare a new piece.

(1.2) Place the 14-in. squares between approximately 1/16-in.-thick aluminum plates which are at least 14-in. in length on a side.

CAUTION: Inspect squares for pinhole tears, overlapped wrinkles, deep scratches or other defects which could allow fluid leakage. Scrap parts having these defects.

(1.3) Place the assembly in a circulating air oven. Set temperature at 66 ± 8°C (150 ± 15°F). After the oven has attained set temperature, hold for 1 hour. Raise oven temperature 56°C (100°F) per hour until 354°C ± 16°C (670°F ± 30°F) is reached. Hold the material at this temperature for a minimum of 30 min.

(1.4) Shut off oven and allow material to cool to near room temperature before removing from the oven.

NOTE: When oven temperature is less than 28°C (50°F) above room temperature, the oven door may be opened to cool the material more quickly.

(1.5) Label each sheet in one corner with the date annealed. If the sheets are not going to be used immediately, store them in an area where they will not be damaged. It is preferable to store them with the 1/16-in. aluminum plates on the outside for protection.

(1.6) Tape one annealed 0.006-in.-thick sheet on the 7.505-in. diam form block.

(1.7) Hand-form and roll the piece of aluminum foil to fit the contour of the form block. The cylindrical section should extend down a minimum distance of 3/4-in. (7/8-in. preferred) from the flange face all around the form block. Label each aluminum foil liner in a corner with the date formed.

(1.8) Select two liners per kit. Label each liner in one corner with kit number.

CAUTION: Inspect foil for defects noted in (1.2). Scrap parts having these defects.

(1.9) Package each kit in a 4-to 8-mil thick polyethylene film bag. Label the bag with kit number, date annealed, and date formed.

(2) Aluminum Liner Component Preparation

(2.1) For each aluminum-lined test cylinder, cut out the liner kit comprised of the quantities and sizes listed in table C-I.

CAUTION: Take extreme care to prevent damage to liner component by wrinkling, indenting, scratching, tearing, puncturing, etc. Always wipe off contacting surfaces when laying foil down on any object. Any damaged liner must be scrapped.

TABLE C-I

ALUMINUM LINER COMPONENTS-0.002-IN. THICK

Quanitity	Size*, in.	Application
1	19-1/4 x 25	Cylindrical liner (Cut using a template. Mark in a notched area the position on the mandrel, e.g., outside large end).

* Shapes and dimensions are shown in figure C-1. The flange liner shown in figure C-1 was prepared as described in (1) above.

(2.2) If etching of the liner components cannot be carried out immediately, place the components between 1/16-in. aluminum plates, package in a 4-to 8-mil-thick polyethylene bag and store in an area in which the components will not be damaged.

CAUTION: When packaging components, inspect per (1.2). Scrap parts having defects noted in (1.2).

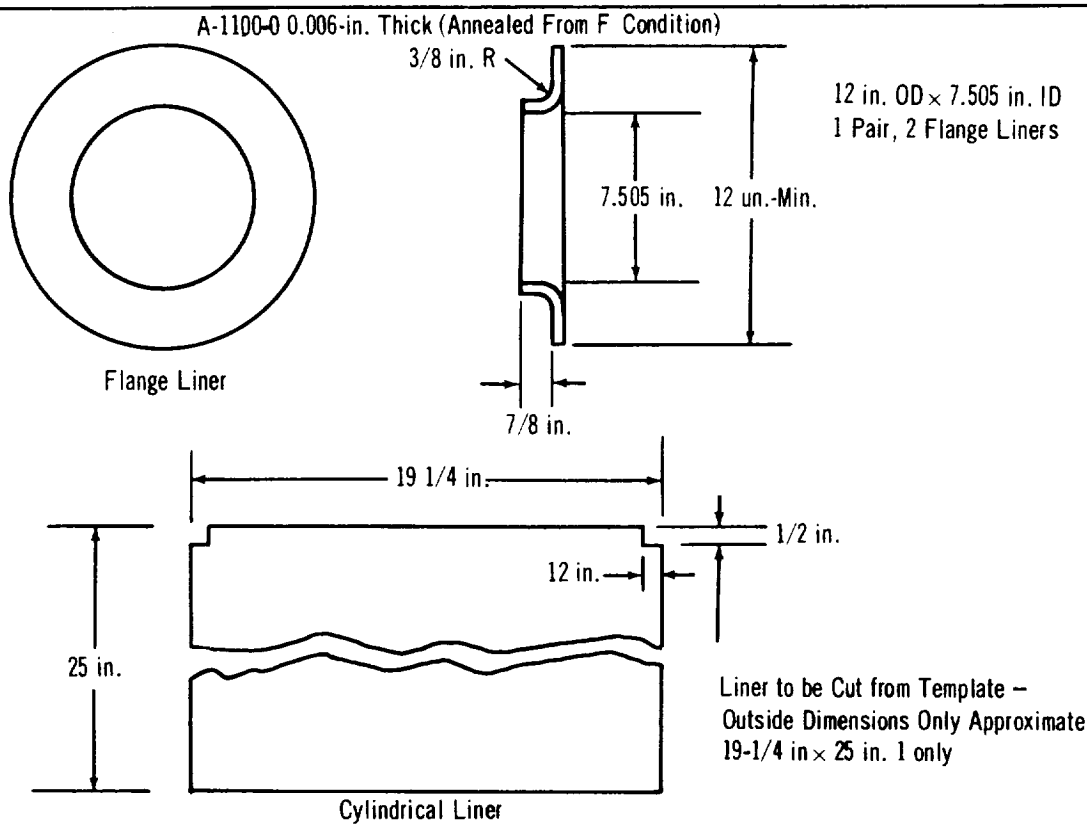


Figure C-1. Aluminum Liner Components

(2.3) Etch the surfaces of the aluminum parts listed in table C-II in the following manner.

(2.3.1) Wipe off all surfaces to be etched with a rag wetted with acetone.

(2.3.2) Allow parts to dry.

(2.3.3) Brush the surface to be etched with Para-Jell 105D* (jelly etch). Agitate jelly with brush every 5 min.

(2.3.4) Thoroughly rinse jelly etch from surfaces with water after 30 $\pm$ 5 min.

(2.3.5) Check water break (water beads should not form on the aluminum surface). If beads form, re-etch surface.

(2.3.6) Test the surface rinse water with universal pH paper. If the pH paper is not the same color as it is in pure tap water, continue to rinse the surface and check the rinse water with pH paper again.

(2.3.7) Shake off surface water and blot with a clean rimple cloth or clean rag until dry, being careful not to damage liner.

* Supplied by Semco Sales and Service.

(2.3.8) Place the etched liner in a circulating air oven at 71°C ± 8°C (160 ± 15°F) for a minimum of 30 min and a maximum of 2 hr.

(2.3.9) Parts must be primed (See Section 3.2) within 8 hr after etching is completed.

TABLE C-II

ETCHED SURFACE DESCRIPTION FOR PARTS

Part	Etched surface description
Flange liners (2)	Complete outside surface including circumferential seam and flange liner where adhesive is applied.
Cylindrical liner (1)	(a) Inside surface; 2-1/2-in. ± 1/2-in. wide strip around all edges. (b) Complete outside surface.
CAUTION: Do not allow contact of the etched surface with the bare skin or any contaminated material during any subsequent handling.	

(2.4) Scrim Cloth Cutting*

(2.4.1.) Cut all scrim cloth parallel with the warp yarns where possible. One or two warp yarns may be pulled from the scrim along the cut line to provide a guide for cutting.

(2.4.2) For each aluminum-lined test vessel, cut out the scrim cloth kit comprised of the shapes and sizes of 112 glass cloth ("E" glass), A-1100 finish as shown in figure C-2.

(2.4.3) Package each kit separately in a 4-to 8-mil thick polyethylene film bag. Label with date of cutting and packaging.

(3) Aluminum Liner Lay-Up

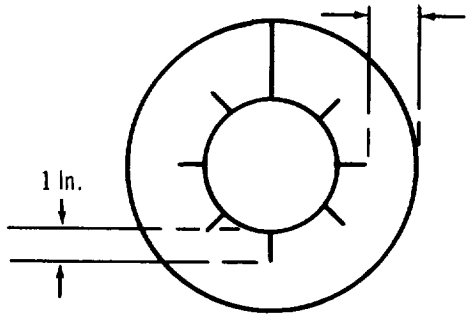
(3.1) Apply a uniform spray coat of GS-3 release agent** to all tools used for liner lay-up and subsequent structure component lay-up which may come in contact with the adhesive or resin.

CAUTION: When using GS-3 aerosol release, be sure that no spray contacts the surfaces or materials which are to be adhesive-or resin-bonded.

* Where applicable.

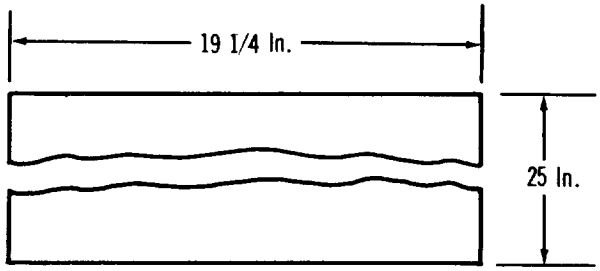
** Manufactured by Ram Chemical Co.

Style 112 E Glass A-1100 Finish 1-7/8 In.



12 In. O.D.
1 Pair, 2 Finger Rings

(a) Flange Liner Scrim



19-1/4 In. x 25 In. 1 Only

(b) Cylindrical Liner Scrim

Figure C-2. Scrim Cloth Size

(3.2) Prime the etched aluminum liner components with 3M silane primer XD3901 (brush application). Prime the surfaces indicated in table C-III as follows:

(3.2.1) The relative humidity of the fabrication area shall be measured before beginning and after completion of the primer and adhesive application. Priming and bonding operations shall be performed only between the range of 30 to 65% relative humidity for maximum primer performance.

(3.2.2) Apply a very thin brush coat (approx. 0.0005-in. thick) of primer. Minimize areas of overlap which receive a repeat brush coat of primer.

WARNING: Avoid inhalation of, or skin contact with primer.

(3.2.3) After priming, let dry for 30 min. minimum.

CAUTION: Bag and seal all primed parts which are not to be used immediately. Use all primed parts within 48 hr of completion of priming. Bare skin or contaminated materials must never contact primed surfaces.

TABLE C-III

AREAS OF PRIMER APPLICATION

Part	Primer surface
Flange liners (2)	The entire surface of the side which has been completely etched.
Cylindrical liner (1)	(a) The outside $\frac{1}{2} \pm \frac{1}{\frac{1}{4}}_{0}$ in. of the two circumferential seam edges and the unnotched longitudinal seam edge. (Do not prime the double notched edge.) (b) The entire etched surface of the opposite side.

(3.3) Install the mandrel shaft assembly in the filament winding machine. Assemble the mandrel with two primed flange liners. Tighten the mandrel drawbar securely. Be sure the contacting surfaces of the mandrel and flange liner are clean and free of any particles. Check the mandrel joints especially at the large end so that the finished part will not contain an undercut in that area. There should be a minimal ridge.

(3.4) Work the flange liners into the mandrel surfaces, particularly at the radii so there is a close fit and no wrinkles.

(3.5) Trim excess foil from outside of flange liners and tape the outsides of liners to the mandrel with cellophane tape to prevent resin incursion between the mandrel and the flange face.

CAUTION: Be sure the tape can be easily removed after lay-up and cure of cylinder. The tape must be removed just before mandrel removal.

(3.6) While the flange liner is tight against the mandrel, trim excess foil from the cylindrical section of the flange liner so that a smooth, even edge is obtained. The distance from the edge to the flange face should be a minimum of 3/4 in., preferably 7/8 in., but no more than 7/8 in., and the same distance ($\pm$ 1/16 in.) all around.

(3.7) To build up a smooth surface on which to wrap the cylindrical aluminum liner, cut a Mylar spacer from 0.002-in.-thick Mylar sheet to fit between the trimmed smooth edges of the flange liners within 1/32 in. of the edge on each end (no overlap). Wrap cylinder three times to match the flange liner thickness.

(3.8) Mark the starting position of the longitudinal edge of the cylindrical aluminum liner as follows:

Wrap a liner template around the Mylar spacer. The longitudinal seam should correspond to the taped Mylar spacer seam. Match the circumferential edges and mark the longitudinal seam position.

(3.9) Verify that the flange liners and cylindrical aluminum liner are free of defects per (1.2).

(3.10) Prepare adhesives to proportions given in Appendix A.

(3.11) The relative humidity of the fabrication area shall be measured before beginning and after completion of the primer and adhesive application. Priming and bonding operations shall be performed only between the range of 30 to 65% relative humidity for maximum primer performance.

(3.12) Squeegee a thin film of adhesive on all faying seam surfaces of the aluminum flange and cylindrical liners except the longitudinal seam on the aluminum liner adjacent to the notched edge.

(3.13) Butt the joint. Position joints so they are displaced a minimum of 2 in. from the longitudinal liner seam.

(3.14) Carefully center the aluminum liner between the flanges (with the completely etched surface out) and tape the notched edge to the Mylar spacer continuously matching the longitudinal liner seam with the marked

position. The liner should be taped so that it can be applied with the winding machine rotating in the same direction as it is during tape winding. The edge of the tape should match the notched edges parallel with the longitudinal liner seam (for masking purposes) and should extend 1/16 in. to 1/8 in. over each of the notched edges and parallel with the circumferential seam to butt against the cylindrical edges of the flange liners (but not over any part of the flange liners).

(3.15) Wipe off all aluminum and Mylar surfaces which will be in contact (except those with adhesive) to remove foreign particles.

(3.16) Finish the cylindrical liner lay-up while jogging the filament winding machine in the direction used for tape winding. The liner lay-up shall be accomplished as follows:

(3.16.1) Carefully lay the cylindrical aluminum liner around the Mylar spacer.

(3.16.2) Work the liner from inside out toward the circumferential seams to smooth and remove slack.

(3.16.3) Press the liner into the circumferential seams progressively to eliminate air.

(3.16.4) When the longitudinal seam area is accessible, squeegee adhesive on the longitudinal seam area adjacent to the taped edge of the liner.

(3.16.5) When all but 1 in. to 2 in. of liner has been applied, trim liner to provide a 1/2 in. overlap seam beyond the taped liner. Complete liner application.

(3.16.6) Squeegee the circumferential seams outward. Smooth and tighten the liner during this operation.

(3.16.7) Squeegee the longitudinal seam pushing air outward. Hold the liner trailing the outside overlap seam while squeegeeing to prevent loosening of the liner.

(3.16.8) Eliminate liner wrinkles by squeegeeing.

CAUTION: During these operations be careful not to tear or stretch the liner out of shape. The thin aluminum foil is very soft and vulnerable to deformation or tearing.

(3.17) Verify (visually) that there is a bead of adhesive along all seam edges. Remove excess adhesive from the edge of the longitudinal seam.

(3.17.1) The vessel fabrication procedures in (4) below, to be described shortly, must be completed within 8 hr. If sufficient time is available, continue to (4). If sufficient time is not available to complete (4), package and store the lay-up as follows:

(3.17.2) Cut one sheet of 5-mil-thick polyethylene film 20 in. x 24 in. for a release sheet. Cut Mylar lag sheet in the same shape as the cylindrical liner except delete the notches. Use 0.005-in.-thick Mylar film.

(3.17.3) Wipe excessive adhesive from the radii.

(3.17.4) Center and wrap the polyethylene sheet around the liner starting with one edge about 1/32 in. from the trailing edge of the liner outside longitudinal seam. Stretch to eliminate wrinkles. Trim the length and butt joint.

(3.17.5) Center and wrap the Mylar lag sheet around the polyethylene release sheet leading the polyethylene joint by about 1-1/2 in. Lap the Mylar joint 1-1/2 in. and trim so the outside edge of the Mylar corresponds to the polyethylene seam and the overlap joint bears on the liner longitudinal seam.

(3.17.6) Lag-wrap the assembly with a 2-in. wide polyvinyl acetate (PVA) tape. Apply with hand tension from the tape roll. Start about 4 in. inside one circumferential seam; change direction and lag-wrap the cylinder and other circumferential seam. Again change direction and lag-wrap the cylinder until the start of the wrap is lapped. Tape down the lag tape with masking tape (DPM 884).

NOTE: When wrapping, butt the edge of the PVA tape but do not allow gaps in any of the first layer of tape.

(3.17.7) Verify (visually) that there is no serious liner wrinkling and that the adhesive has beaded at visible edges of all seams.

(3.17.8) Bag the lay-up with polyethylene bag and seal with masking tape. Leave at ambient temperature if exposure will not exceed 24 hr; otherwise, store at -18°C to -12°C (0°F to 10°F).

CAUTION: Be certain the bag is sealed before storing.

(4) Structural Component Lay-Up

(4.1) If the partial lay-up has been stored in a refrigerator, thaw it in a 49° ± 8°C (120° ± 15°F) oven and install in the filament winding machine.

CAUTION: Do not break end seal until the material reaches a temperature sufficient to prevent water condensation on the part.

(4.2) Remove all wrapping material.

CAUTION: Do not stretch the liner while removing the lag tape.

(4.3) Squeegee the flange liners into the mandrel radii to assure full radii with no voids or wrinkles.

(4.4) Prepare adhesive to specified proportions and impregnate the liner scrim.

(4.5) Center and lay up the impregnated scrim on the liner. Offset the cylindrical scrim longitudinal joint from the liner joint 3 in. to 4 in. Trim the scrim to make a butt joint. Squeegee the scrim in the following manner. Squeegee a portion of adhesive evenly over a area of Mylar film corresponding to the cloth size. Lay the scrim cloth over the adhesive on the Mylar and allow the adhesive to wet and soak the scrim thoroughly. Apply and squeegee adhesive over any dry spots. Squeegee the excess adhesive from the scrim just before use.

(4.6) Squeegee a generous thickness (approximately 20 to 30 mils) of adhesive onto the radii and on the flange liners. Cover surfaces as evenly as possible.

(4.7) Lay up the impregnated flange liner scrim cloth. Offset the joints 5 in. to 6 in. from cylindrical scrim joint. Squeegee the scrim.

(4.8) Wrap layer of hoop wraps directly over the adhesive system.

(4.8.1) Tie the tape down with one complete wrap placed 1-1/8 in. to 1-3/8 in. from the flange face at the small diameter end.

NOTE: References to the flange face with respect to distances always mean the flange liner face, not the fiberglass lay-up.

(4.8.2) Wrap toward the small diameter flange, gradually increasing the tension until, at a position adjacent to the radius, the tension is 20 lb.

(4.8.3) Disengage the carriage travel and wind one complete turn.

(4.8.4) Reverse the former carriage direction and engage the carriage. Wrap one layer to the radius of the flange face of the large diameter end. When winding hoops use a heat gun, if necessary, to obtain butted adjacent hoop wraps (or a minimum gap) and good flow. A small amount of squeegeeing of hoops is permissible to eliminate gaps and overlap and to remove excess resin and/or adhesive.

NOTE: Hold gaps in the test section (middle 9 in. of cylinder) to 0.010 in. or less. There is no limitation in buildup areas. Overlaps in the test section are not allowed unless they can be eliminated by squeegeeing.

(4.8.5) Disengage the carriage when the tape is adjacent to radius and wrap one complete turn.

(4.8.6) Reverse former carriage direction. Engage carriage and wrap 1-in. from the flange face.

(4.8.7) Release the tension and wrap one-half turn.

(4.8.8) Cut the tape and smooth it into previous winding.

(4.8.9) Measure width of 10 windings of tape in the test section to the nearest 1/32 in.

CAUTION: Keep the tensioner and payoff rolls clean so the tape will not wander or jerk during winding operation.

(4.9) Squeegee the flange liner scrim to fit against the flange liners smoothly. Squeegee the test section to smooth adhesive bleed-through.

NOTE: It is possible to stop anywhere in the process of structural component lay-up from this point on. If work is stopped for more than 2 hr, the part must be bagged and sealed and stored in a -18°C to -12°C (0°F to 10°F) refrigerator. When work is to be resumed, thaw the part in an oven at 49 $\pm$ 8°C (120 $\pm$ 15°F).

(4.10) Thaw the longitudinal reinforcement.

(4.11) Weigh out in separate containers, 100 g of Epi-Rez 5101 and 15 g of APCo 322. Heat the containers in an air circulating oven at 49°C $\pm$ 6°C (120°F $\pm$ 10°F) for 20 $\pm$ 5 min. Thoroughly mix the hot resin and hardener.

CAUTION: The working life of this resin system is 1-1/2 to 2 hr.

(4.12) Generously coat with resin the flange faces, radii, and a portion of the cylinder on each end extending 3 in. to 3-1/4 in. from each flange face.

NOTE: Unless otherwise noted herein, application of resin on the cylinder during the entire lay-up should be accomplished all the way around the cylinder to the previously applied glass cloth.

(4.13) Lay-up one circular donut on top of the flange liner scrim.

(4.14) Center the lay up the full length (23-1/2 in. wide) longitudinal reinforcement. Squeegee and remove backing material. Squeegee the fingers into the radii and onto the faces of the flanges. Butt joint the longitudinal reinforcement at the large diameter end. There will be a small overlap in the seam at the small diameter end. Squeegee the over-lap as smooth as possible.

CAUTION: Do not cut any longitudinal reinforcement filaments. Slit between filaments.

(4.15) Advance reinforcement on large diameter by wrapping with one layer of PVA tape to prevent expansion of the laminate and then heating area with heat gun for 1 hr. Remove PVA tape upon completion of heating.

(4.16) Wrap the second hoop wrap exactly as the first hoop wrap except start wrap near the large diameter end and initially wrap toward the large diameter end. After completion of wrap, measure the width of 10 windings in the test section.

CAUTION: Avoid longitudinal displacement. Maximum allowable is 1/8 in.

(4.17) Thaw a prepreg cloth kit.

(4.18) Prepare 115 g of resin per (4.11) above and generously coat with resin the flange, faces, radii, and a portion of the cylinder on each end extending 4-1/2 in. to 4-3/4 in. from each flange face.

(4.19) Lay up the first half of the impregnated cloth for the flange and buildup.

NOTE: Minimize voids and bridges. Compact the lay-up, especially at the radii. Use a heat gun, if necessary, but minimize the resin advancement so the resin remains sticky.

(4.20) Tie down the buildup on each end with the first two-layer shingle tape wrap as follows.

(4.20.1) Tie the tape down with one complete wrap 1-1/2 in. to 1-3/2 in. from flange face.

(4.20.2) Wrap toward flange face with machine lead exactly as the first hoop wrap.

NOTE: Smooth the shingle tape wrap while wrapping to provide a smooth surface for the longitudinal reinforcement lay-up. If a depressed area results during the wrap, disengage the carriage so that the tape wraps continuously in the depressed area until built up til smooth. Heat may be used to effect resin flow and to aid in smoothing the wrapped surface.

(4.20.3) When the tape wrap reaches a flat surface (no slope), increase the tension gradually until at a position adjacent to the radius the tension is 40 lb.

(4.20.4) Disengage the carriage and wrap one full turn.

(4.20.5) Reverse the former carriage direction and wrap 2-3/4 in. to 3-1/4 in. from the flange face.

(4.20.6) Reverse the former carriage direction and wrap about 2-in. (1 in. from flange face).

(4.20.7) Release tension and wrap one-half turn. Cut the tape and smooth into the previous winding.

(4.21) Lay up the second half of the impregnated cloth for the flange and end buildup.

(4.22) Tie down the buildup on each end with the second two-layer shingle tape wrap as follows:

(4.22.1) Tie tape down with one complete wrap 2-in. to 2-1/4 in. from flange face.

(4.22.2) Wrap towards flange face with the machine lead identical to that for the second hoop layer.

(4.22.3) When the tape wrap reaches a flat surface (no slope), increase the tension gradually to 20 lb.

(4.22.4) Disengage carriage and wrap one full turn.

(4.22.5) Reverse former carriage direction and wrap to approximately 4-1/2 in. from the flange face.

(4.22.6) Reverse former carriage direction and wrap approximately 3-in. (i.e., 1-1/2 in. from the flange face).

(4.22.7) Release the tension and wrap one-half turn.

(4.22.8) Cut tape and smooth into previous winding.

(4.23) Generously coat the flange, end buildups, and radii with resin.

(4.24) Center and install clamp rings on each end to pressure flange lay-up and radii. Tighten clamp rings so there is no gap at the clamp ring joints.

NOTE: A heat gun may be used to effect softening and flow. If clamp ring clearance from the glass around the radius is greater than 1/32 in. all the way around, remove the clamp rings and wrap prepreg tape around radius area to eliminate this condition.

CAUTION: Be careful that the vessel is not damaged by the clamp rings during installation.

(4.25) Install clamp ring clamps and evenly tighten to finger tightness to obtain an even flange thickness.

(4.26) Preheat a circulating air oven to 66° ± 6°C (150° ± 10°F).

(4.27) Remove the part from the filament winding machine. Attach thermocouple and strip chart temperature recorder. Place the part in oven at 66° ± 6°C (150 ± 10°F). Record on the chart the date and time of start of cure, model and serial numbers of the part, and length of previous exposure at room temperature and refrigerated temperature.

(4.28) Cure the part 1 hr at 66° ± 6°C (150° ± 10°F). During this time, tighten the clamp ring clamps evenly every 15 min. Tighten only finger tight to obtain an even flange thickness.

(4.29) Increase oven temperature to 121° ± 8°C (250° ± 15°F) and cure for 8 hr. No rotation is necessary.

NOTE: If the strip chart temperature recorder is not available, record the time when the elevated cure temperature is attained and oven temperature every hour of the cure.

(4.30) Turn off oven at the end of the cure cycle and remove part from the oven.

(4.31) Remove clamp ring clamps and clamp rings.

CAUTION: Be careful that the vessel is not damaged before, during, or after clamp ring removal.

(4.32) Remove mandrel.

(5) Test Flange Bonding

(5.1) The test group shall drill bolt holes in the cylinder flanges in the appropriate areas for test flange attachment.

(5.2) Make sure all necessary metal parts (two test flanges, two deflection wire brackets, eight backup ring half-sections and a sufficient number of nuts, bolts and washers) are available for bonding test flanges to the seal rings or liner of the test cylinder. Verify they are clean and free of adhesive.

(5.3) Apply GS-3 release agent to all components noted above except the test flanges. Do not apply release agent to any portion of the test cylinder.

(5.4) Cut two 1/2-in. x 36-in. strips of scrim cloth from 112 glass cloth ("E" glass), A-1100 finish.

(5.5) Wipe all faying surfaces with a clean cloth dampened with acetone and let dry for 15 min.

(5.6) Mix a kit of the two component adhesive system, L-100/MOCA, and apply a generous amount on one cylinder flange or seal ring face.

(5.7) Lay up on the cylinder flange or seal ring face one 1/2-in. wide scrim strip inside but adjacent to the bolt holes. Butt the strip and trim excess; squeegee; leave a generous amount of adhesive.

(5.8) Apply a thin film of adhesive to the faying surface of the test flange so the surface is completely wetted.

(5.9) Position the test flange using two bolts through the test flange about 180° apart for guides. Place the test flange against the scrim adhesive so there will be a minimum of side movement necessary to insert all the flange bolts.

(5.10) Install backup rings, deflection wire brackets, bolts, washers, and nuts. Tighten nuts finger tight and allow 5 min before continuing.

(5.11) Torque nuts to 20 $\pm$ 1 lb-ft. Make sure there is a bead of adhesive around the outside of the test flange-test cylinder joint.

(5.12) Repeat (5.6) through (5.11) for the other end of the test flange. Make sure there is no foreign material inside vessel before bonding.

(5.13) Cure 24 hr minimum at ambient temperature of 18°C (65°F) minimum.

APPENDIX D

TEST PROCEDURE

General

Test vessels, burst and cyclic, are instrumented with four longitudinal deflection gages and two circumferential growth deflection gages (where applicable) to permit two longitudinal and circumferential strain measurements. The internal pressure of the burst vessels is increased until failure occurs. The pressure is applied at a rate that causes the vessel to strain at a rate equal to, or less than, 1%/min. During the test, continuous recordings are made of internal pressure, longitudinal deflection, and circumferential growth (where applicable). For all cyclic tests, a cycle is defined as (1) pressurizing from minimum transfer line pressure to the internal pressure corresponding to 2.0% strain, which is identified from the applicable temperature burst test, and then (2) depressurizing to the minimum line pressure.

A detailed description of the test procedure is given in the following paragraphs.

Ambient Temperature Test. - The procedure for ambient temperature testing is as follows:

(1) Prepare specimen for test.

(A) Drill $\frac{33}{64}$ in. diam bolt holes in both specimen flanges using drilling template.

(B) Install inlet and outlet flanges on specimen using rubber gasket, NAS 1008-34A bolts and 119 FW nuts. Torque to 40 ft-lb.

(C) Leak check specimen and flange seals with ambient temperature helium at 50 psi.

(2) Install specimen in test fixture.

(A) Install and position specimen in vacuum chamber.

(B) Install specimen outlet line adapter flange using NAFLEX spacer flange seal P/N VD 261-0036-0009.*

(C) Bolt retainer ring to vacuum chamber inlet port flange.

* Manufactured by Navan Products Corp.

(D) Connect deflection transducer tie wires to specimen.

(E) Install specimen centering device.

(F) Connect transfer line from water reservoir to specimen inlet line.

(3) Pretest procedure.

(A) Open specimen isolation valve.

(B) Fill specimen and reservoir with water then close fill and bleed valves.

(C) Set up instrumentation and calibrate pressure transducers with known pressures.

(D) Turn on control panel power.

(E) Check actuation of vent valve and motorized pressure regulator.

(F) Check recorder for sufficient recording paper.

(4) Burst Test.*

(A) Close vent valve.

(B) Open helium supply shutoff valve.

(C) Start recorders.

(D) Control rate of pressure rise in specimen by varying applied voltage to motorized pressure regulator.

(E) When specimen fails close helium supply shutoff valve.

(F) Turn off recorders.

(G) Return motorized regulator to closed position.

(H) Turn off control panel power.

(5) Cyclic Test.*

(A) Close vent valve.

(B) Open helium supply shutoff valve.

* Depending on whether burst or cyclic test.

(C) Start recorders.

(D) Control rate of pressure rise in specimen by varying applied voltage to motorized pressure regulator.

(E) When specimen pressure reaches desired value, depressurize specimen to zero by opening the vent valve and closing the supply shutoff valve.

(F) Close motorized regulator.

(G) Repeat three steps above for applicable number of cycles or until specimen fails.

(H) After the last cycle close helium supply shutoff valve.

(I) Turn off recorders.

(J) Return motorized regulator to closed position.

(K) Turn off panel power.

(6) Removal of specimen from test fixture.

(A) Close specimen isolation valve.

(B) Drain water from specimen.

(C) Disconnect water transfer line from specimen inlet line.

(D) Remove specimen centering device.

(E) Disconnect deflection gage tie wires from specimen.

(F) Remove retainer ring from vacuum chamber inlet port flange.

(G) Remove specimen from vacuum chamber.

(H) Leak check specimen and flange seals with ambient temperature helium at 50 psi (where applicable).

(I) Photograph and examine specimen.

(J) Analyze mode of failure.

Cryogenic Temperature Test (-432°F). - The procedure for cryogenic temperature testing is as follows:

(1) Prepare specimen for test.

(A) Drill $\frac{33}{64}$ in. diam bolt holes in both specimen flanges using drilling template.

(B) Bond inlet and outlet flanges to specimen using polyurethane-fiberglass cloth (see Appendix C), NAS 1008-34A bolts, and 119 FW nuts. Torque to 80 ft-lb after cuving for 24 hr.

(C) Leak check specimen and flange seals with ambient temperature helium at 50 psi.

(2) Install specimen in test fixture.

(A) Install "O" ring (AN 6227B-49) on vacuum chamber inlet port flange.

(B) Install and position specimen in vacuum chamber.

(C) Install specimen outlet line using NAFLEX spacer flange seals P/N VD 261-0036-0009.

(D) Bolt retainer ring to vacuum chamber inlet port flange.

(E) Connect deflection gage tie wires to specimen.

(F) Install specimen centering device.

(G) Connect bleed lines to the bleed ports of the flanges using the NAFLEX spacer flange seal.

(H) Leak check the bleed lines with ambient temperature helium at 50 psi.

(I) Connect liquid transfer line to specimen inlet line and insulate joint.

(J) Make electrical check on deflection transducers.

(K) Install cover on vacuum chamber.

(L) Start vacuum pumps to evacuate chamber and bleed line from NAFLEX seals.

(M) Open vacuum pump shutoff valves.

(3) Pre-run procedure.

(A) Turn on control panel power.

(B) Open Pirani gage transducer shutoff valve.

(C) Transfer control of vacuum pump shutoff valve from 115 Vac to the instrument recording the Pirani gage output.

(D) Set up instrumentation and calibrate pressure transducers with known pressures.

(E) Open vacuum chamber pressure transducer shutoff valve.

(F) Check actuation of all valves.

(G) Close bleed line vacuum shutoff valve.

(H) Make electrical connection to liquid hydrogen pump.

(I) Adjust liquid hydrogen pump outer chamber purge valve for proper bleed.

(J) Adjust liquid hydrogen pump motor purge to 1 psi.

(K) Check recorders for sufficient recording paper.

(4) Cyclic test.*

(A) Pressurize liquid hydrogen storage tank to 50 psi.

(B) Close specimen fill valve.

(C) When pressure in specimen has bled to zero, run recorders long enough to obtain cold-zero position of deflection gages (where applicable).

(D) Open fill valve.

(E) When specimen is again filled with liquid, start recorders.

(F) Close main vent valve.

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* Depending on whether burst or cyclic test.

(G) Close liquid hydrogen pump by-pass valve.

(H) Control rate of pressure rise in specimen by means of the throttle vent valve.

(I) When specimen pressure reaches desired level, decrease pressure to the minimum transfer line pressure.

(J) Open pump by-pass valve.

(K) Open main vent valve.

(L) Repeat seven immediate steps above for applicable number of cycles or until specimen fails.

(M) After last cycle, turn off control panel power which will automatically shut down system to a safe condition.

(N) Close liquid hydrogen supply shutoff valve.

(O) Turn off liquid hydrogen pump.

(P) Turn off recorders.

(Q) Return control panel switches to their normal position.

(5) Post-test procedure.

(A) When liquid hydrogen has dissipated, turn off carbon dioxide purge to burst chamber.

(B) Turn off liquid hydrogen pump motor purge and outer chamber purge.

(C) Disconnect liquid hydrogen pump from power source.

(6) Chilldown procedure.

(A) Turn on burst chamber carbon dioxide purge.

(B) Open liquid hydrogen supply shutoff valve.

(C) Open main vent and throttle vent valves.

(D) Open specimen fill valve.

(E) Open pump by-pass valve.

(F) Adjust liquid hydrogen pump inlet chamber bleed valve to give a slight bleed of liquid to atmosphere.

(G) When pump has chilled sufficiently, start pump.

(H) When entire system has chilled sufficiently, proceed with test.

(7) Burst Test.*

(A) Pressurize liquid hydrogen storage tank to 50 psi.

(B) Close specimen fill valve.

(C) When pressure in specimen has bled to zero, run recorders long enough to obtain cold-zero position of deflection transducers.

(D) Open fill valve.

(E) When specimen is again filled with liquid start recorders.

(F) Close main vent valve.

(G) Close liquid hydrogen pump by-pass valve.

(H) Control rate of pressure rise in specimen by means of the throttle vent valve.

(I) When specimen fails, turn off control panel power which will automatically shut down system to a safe condition.

(J) Close liquid hydrogen supply shutoff valve.

(K) Turn off liquid hydrogen pump.

(L) Turn off recorders.

(M) Return control panel switches to their normal position.

(N) Turn off vacuum chamber vacuum pump.

(O) When system has warmed to ambient temperature, secure control panel.

(8) Removal of specimen after test.

(A) Remove cover from vacuum chamber.

(B) Disconnect liquid transfer line from specimen inlet line.

(C) Disconnect bleed lines from flanges using the NAFLEX spacer flange seal.

(D) Remove specimen centering guide.

(E) Disconnect deflection gage tie wires from specimen.

(F) Remove specimen outlet line.

(G) Remove retainer ring from vacuum chamber inlet port flange.

(H) Remove specimen from vacuum chamber.

(I) Leak check specimen and flange seals with ambient temperature helium at 50 psi (when applicable).

(J) Photograph and examine specimen.

(K) Analyze mode of failure.

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